PRODUCTION AND CHARACTERIZATION OF ENVIRONMENTALLY FRIENDLY OIL BASE MUD FROM RAPHIA PALM SEEDS (*RAPHIA HOOKERI*)

BY

OLASOJI OLUWABORI CHRISTIANAH
19/ENG07/016

PETROLEUM ENGINEERING PROGRAMME
DEPARTMENT OF CHEMICAL AND PETROLEUM ENGINEERING
COLLEGE OF ENGINEERING
AFE BABALOLA UNIVERSITY, ADO-EKITI, EKITI STATE, NIGERIA

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IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF THE DEGREE OF THE BACHELOR OF ENGINEERING (B.ENG) DEGREE IN PETROLEUM ENGINEERING

JUNE 2024

DECLARATION

I, OLASOJI Oluwabori Christianah (19/ENG07/016), hereby declare that this project work titled "Production and Characterization of Environmentally friendly Oil base mud from Raphia Palm seeds(*Raphia Hookeri*)", carried under the supervision of Engr. A.T. Ogunyemi and submitted in partial fulfillment of the requirements for the award of a Bachelor of Engineering (B.Eng.) Degree in Petroleum Engineering, is my original work and has not been presented for any degree elsewhere, to the best of my knowledge. All sources of information are specifically acknowledged using references.

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OLASOJI Oluwabori Christianah　　　　　　　　　　DATE

CERTIFICATION

This is to certify that this research project titled "Production and Characterization of Environmentally Friendly Oil Base Mud from Raphia Palm Seeds (*Raphia Hookeri*)" was carried out by OLASOJI Oluwabori Christianah with the matriculation number 19/ENG07/016 and that the report was submitted to the Department of Chemical and Petroleum Engineering, College of Engineering, Afe Babalola University, Ado-Ekiti.

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DEDICATION

I dedicate this research project to the Almighty God for his favour, grace and guidance to complete this project and my academic pursuit. I am also dedicating it to myself for the constant discipline and motivation I instilled on myself. Finally, to my family and friends for their support and encouragement.

ACKNOWLEDGEMENT

Many people have been of assistance to me, during my academic pursuits. It is impossible to list everyone here; however, because of the importance of their contributions, I wish to express my thanks and appreciation to some very special people.

With a deep sense of appreciation, respect, and gratitude, I want to say thank you to my family for their care and support toward the completion of this program, from the beginning of my pursuit for a B.Eng. degree in Petroleum Engineering to this point.

Many thanks to my supervisor, Engr. T. A Ogunyemi for the extra help, insightful comments and guidance concerning this project, Engr. Mrs. Gbemi for sticking with me and constantly being of help. Also, the Head of the Chemical/Petroleum Engineering Department, Engr. Dr. Adesina, who has so graciously drawn from his experience in academia and industry to counsel and guide me through my undergraduate petroleum engineering studies. I also take this opportunity to express a deep sense of gratitude to all my lecturers for their enthusiastic encouragement and the valuable information provided by them in their respective fields.

To my family, I’m indeed grateful to my mom, Funmi, Yemi and my dad in his blessed memories, who encouraged me to study engineering against all odds, love you dad. To my slats: Favour, Sahadat, Rebecca, Rayyan, and all my amazing classmates, thank you for 5 years of beautiful experiences.

Finally, my greatest appreciation goes to Almighty God for his consistent and unwavering grace and mercy all my life.

ABSTRACT

This research project aims to investigate the feasibility and sustainability of producing an environmentally friendly oil base mud from Raphia palm seeds. The study will involve the entire process from seed selection to mud characterization, emphasizing the potential benefits and applications in the oil and gas drilling industry. Due to the increase in environmental issues, there is a need for drilling companies to come up with a safer, environmentally friendly oil-based drilling fluid. For this study, oil extracted from Raphia Palm seed was used as the base fluid for drilling mud samples in the laboratory. The Raphia Palm seed oil (RPSO) extracted was tested and compared with the standard requirements for oil needed for oil-based drilling fluid. Results showed that the RPSO met the requirements and can be used in formulation of oil-based drilling muds. Oil-in-water emulsion was made using an oil/water ratio of 70 to 30. 254ml of oil and 105 ml of water. 35g of bentonite and barite were finally added to build up the density to 8.58ppg. The mud sample was formulated with Raphia Palm seed oil (RPSO) as the base fluid and compared to a diesel-based mud sample of the same quantity. Different mud tests such as toxicity, filtration, pH, viscosity and density were carried out on each of the samples to ascertain the suitability of their properties for drilling operation and their degree of safety to the environment. The results obtained showed that Raphia Palm seed oil (RPSO) mud has the lowest viscosity (their values) which implies less resistance to flow and lower pressure losses. The outcome of the toxicity test confirmed Raphia Palm seed to be safer and less harmful while diesel is highly toxic as expected. The overall result obtained from the test indicates that Raphia Palm seed oil (RPSO) based muds stand a chance of being among the technically and environmentally viable replacement for the conventional diesel oil-based muds. The study serves as one of the solutions to the environmental problem associated with oil-based drilling operations. It can be adduced from these results that, with appropriate and adequate additives, the formulated synthetic-based drilling mud could compete favorably with bentonite as a possible drilling fluid for the oil and gas industry.

Keywords: Oil-base mud, drilling fluids, environmentally friendly drilling fluid, rheological properties, physicochemical properties, synthetic-based drilling fluid, API standards, additives, bentonite mud, density, pH, pollution, disposal, Emulsification, Raphia palm seeds (ester oil).

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NOMENCLATURE

API- American Petroleum Institute

APEOs- Alkylphenol ethoxylates

BHT- Bottom Hole Temperature

BHA- Bottom Hole Assembly

EPA- Environmental Protection Agency

EDX- Energy Dispersive X-Ray Analysis

FAME- Fatty Acid Methyl Ester

FFA- Free Fatty Acid

FTIR- Fourier Transform Infrared Spectroscopy

GC/MS- Gas Chromatography/Mass Spectrometer

HSE- Health Safety and Environment

HTHP- High Temperature High Pressure

IFT- Interfacial Tension

LCMs- Lost circulation materials

LPLT- Low Temperature Low Pressure

OBM- Oil Base Mud

PV- Plastic Viscosity

ppg- Pounds per Gallon

rpm- Revolutions per Minute

RPSO- Raphia palm seed oil

SBM- Synthetic Based Muds

SEM- Scanning Electron Microscopy

SDGs- Sustainable Development Goals

SFT- Surface Tension

SP- Surfactant/Polymer

TOC- Total Organic Carbon

O/W- Oil-in-water

WBM- Water Base mud

W/O- Water-in-oil

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background of Study

Drilling fluids are used in the oil and gas industry for the drilling of boreholes and construction of oil wells. They are sometimes referred to as “drilling muds" and generally viewed as the “blood” of all drilling operations in the petroleum industry. Drilling mud is typically classified as either water-based or oil-based/invert emulsions depending on the chemical composition. While water-based muds are generally used in many parts of the world in shallow wells and often in shallower portions of deeper wells, the performance of this class of drilling fluid is often poor in extended reach wells. The reasons being, for the most part, the drilling pipe can become stuck in the hole causing delays in drilling, and loss of time and money. Cameron *et al*. (2004) maintain that oil-based drilling fluids have for many years been the drilling fluid of choice for use in challenging borehole sections. This is because oil-based drilling fluids have many advantages including, increased lubricity, optimal shale stability, enhanced shale inhibition, low torque and drag resistance to contamination, high drilling rates and greater cleaning abilities with less viscosity. However, some of the challenges of utilizing an invert emulsion drilling fluid have been the increasing level of environmental concern and legislation associated with their use. So far, another class of drilling fluid has been developed to replace the traditional oil with synthetic organic liquids designed to improve environmental performance. Within the last decade, the oil and gas industry has introduced synthetic-based drilling fluids as substitutes for oil-based fluids because of the widely recognized problem with oil-based mud. A case in point, site discharge of drill cuttings from drilling rigs produced from wells using oil-based muds was banned in the early 1990s as a result of environmental concerns (Story and Lee, 2001). Governments and environmentalists across the globe have welcomed proposals to formulate alternative muds based on emulsions and polymers of various kinds that could do the essential duties of oil-based fluids without causing adverse effects on the environment.

1.1.1 Drilling Fluids

The term drilling fluids or drilling muds generally applies to fluids used to help maintain well control and remove drill cuttings (rock fragments from underground geological formations) from holes drilled in the earth. Drilling fluids, also known as drilling mud, are specialized fluids used to aid in the drilling of boreholes into the earth. They play a crucial role in oil and natural gas exploration and production, as well as in various geotechnical applications. Drilling fluids, also known as drilling mud, are specialized fluids used to aid in the drilling of boreholes into the earth. They play a crucial role in oil and natural gas exploration and production, as well as in various geotechnical applications. The primary functions of drilling fluids are:

1. Hydrostatic Pressure Control: Drilling fluids exert hydrostatic pressure against the walls of the wellbore, counteracting formation fluids and preventing them from entering the well. This is essential for maintaining well control and preventing blowouts.

2. Cuttings Removal: Drilling fluids carry drill cuttings, the rock chips and fragments produced by the drill bit, up to the surface. This helps to keep the wellbore clean and prevent the accumulation of cuttings that could hinder drilling operations.

3. Lubrication and Cooling: Drilling fluids lubricate the drill bit and drilling string, reducing friction and wear. They also provide cooling to the drill bit and surrounding formations, preventing overheating and potential equipment damage.

4. Wellbore Stability: Drilling fluids maintain wellbore stability by supporting the walls of the wellbore and preventing collapse or sloughing. This is particularly important in unconsolidated or fractured formations.

5. Suspension of Cuttings: When drilling operations are paused, drilling fluids suspend drill cuttings in the wellbore, preventing them from settling and potentially causing blockages.

6. Transmission of Hydraulic Power: Drilling fluids transmit hydraulic power to downhole tools and the drill bit, enabling them to function effectively.

7. Information Gathering: Drilling fluids can provide valuable information about the formations being drilled, such as the type of rock, porosity, and permeability. This information is crucial for optimizing drilling operations and evaluating potential hydrocarbon reservoirs.

Considering each of the uses, the primary use of drilling fluids is to conduct rock cuttings within the well. If these cuttings are not transported up the annulus between the drill string and wellbore efficiently, the drill string will become stuck in the wellbore. The mud must be designed such that it can carry the cuttings to surface while circulating, suspend the cuttings while not circulating, and drop the cuttings out of suspension at surface.

The hydrostatic pressure exerted by the mud column must be high enough to prevent an influx of formation fluids into the wellbore, but the pressure should not be too high, as it may fracture the formation. The instability caused by the pressure differential between the borehole and the pore pressure can be overcome by increasing the mud weight. The hydration of the clays can only be overcome by using non-water-based muds, or partially addressed by treating the mud with chemicals which will reduce the ability of the water in the mud to hydrate the clays in the formation. These muds are known as inhibited muds. While drilling, the rock cutting procedure generates a lot of heat which can cause the bits, and the entire BHA (Bottom Hole Assembly) wear out and fail, and the drilling muds help in cooling and lubricating the BHA. These fluids also help in powering the bottom hole tools. In cementing operations, drilling fluids are used to push and pump the cement slurry down the casing and up the annular space around the casing string in the hole.

The drilling fluid must be selected and or designed so that the physical and chemical properties of the fluid allow these functions to be fulfilled. However, when selecting the fluid, consideration must also be given to:

1. The environmental impact of using the fluid
2. The cost of the fluid
3. The impact of the fluid on production from the reservoir

1.1.2 Types of Drilling Fluids

Drilling fluids are classified according to the continuous phase:

1. The WBM (Water Based Muds), with water as the continuous phase.
2. The OBM (Oil Based Muds), with oil as their continuous phase.
3. The Pneumatic fluids (with gases or gas-liquid mixtures as their continuous phase)

This research project narrows focus to oil-based drilling fluids (OBM).

In general, OBM are drilling fluids that have oil as their dominant or continuous phase. A typical OBM has the following composition:

Clays and sand about 3%, Salt about 4%, Barite 9%, Water 30%, Oil 50-80%.

OBM has a whole lot of advantages over the conventional WBM. This is due to the various desirable rheological properties that oils exhibit. Since the 1930s, it has been recognized that better productivity is achieved by using oil rather than water as the drilling fluid. Since the oil is native to the formation it will not damage the pay zone by filtration to the same extent as would a foreign fluid such as water. OBM can be reused, and can also be stored for a long time since microbial activity is suppressed.

The basic kind of oil used in formulating OBM is diesel oil, which has been in existence for a long time, but over the years, diesel oil-based muds have posed various environmental problems.

Water-based muds (WBMs) are usually the mud of choice in most drilling operation carried out in sandstone reservoir, however some unconventional drilling situations such as deeper wells, high temperature/pressure formation, deep water reservoir, alternative shale-sand reservoir and shale resource reservoir require use of other mud systems such as oil-based mud to provide acceptable drilling performance.

OBM is needed where WBM cannot be used, especially in hot environments and salt beds where formation compositions can be dissolved in WBM. OBM have oil as their base and therefore are more expensive and require more stringent pollution control measures than WBM. It is imperative to propagate the use of environmentally friendly and biodegradable sources of oil to formulate our OBM, thereby making it less expensive and environmentally safe and equally carry out the basic functions of the drilling mud such as maintenance of hydrostatic pressure, removal of cuttings, cooling and lubricating the drill string and also to keep newly drilled borehole open until cementing is carried out.

Figure 1.1: A well formulated Drilling mud

1.1.3 Composition of Drilling Fluids

The composition of drilling fluid varies depending on the type of fluid, the specific application, and the desired properties. However, the general components of drilling fluids include:

1. **Base Fluid**

The base fluid is the primary liquid component of the drilling fluid. It provides the fluid's overall volume and viscosity. Common base fluids include:

a. Water: Used in water-based muds (WBMs).
b. Oil: Used in oil-based muds (OBMs).
c. Air or gas: Used in pneumatic fluids.

2. **Solids**

Solids are added to drilling fluids to enhance their properties, such as viscosity, density, and filtration control. Common solids include:

a. Clays: Bentonite clay is the most common clay used in WBMs, providing viscosity and gel strength. Other examples are: Kaolinite, Attapulgite, and Illite
b. Weighting Materials: Barium sulfate (barite) is the most common weighting material, increasing the density of the drilling fluid to counteract hydrostatic pressure. Other examples are Hematite (Fe_2O_3), Ilmenite ($FeTiO_3$), and Siderite ($FeCO_3$)
c. Fillers: Calcium carbonate (chalk) or hematite are used as fillers to adjust the rheology of the drilling fluid. Other examples are: Gilsonite, Silica flour, Asphaltum, and Graphite.

3. Additives

Additives are specialized chemicals added to drilling fluids to achieve specific performance requirements. Common additives include:

a. Viscosity Modifiers: Polymers or clays are used to increase viscosity and improve hole cleaning. Examples are: Organophilic clays, Alkyl ammonium salts, and Fatty acid esters.
b. Filtration Control Agents: Starch or cellulose polymers are used to reduce filtration rate and prevent fluid loss into formations. Examples are: Asphaltenes, Gilsonite, and Polymers
c. Inhibitors: Chemicals added to prevent corrosion, prevent shale hydration, or control formation fluids. Examples are: Clay stabilizer, Emulsion stabilizer, and Scale inhibitor
d. Surfactants: Chemicals added to reduce surface tension, improve wettability, and enhance emulsification. Examples are: Alkylphenol ethoxylates (APEOs) Alkyl amines Fatty acid esters
e. Lubricants: Graphite or molybdenum disulfide are added to reduce friction and wear on drilling equipment.
f. Alkalinity Control: Lime is used to maintain the alkalinity of oil muds at an acceptable level. A high pH (8.5-10.0) is needed to control corrosion and to obtain the best performance from the emulsifiers.
g. Density Control: API barite is the main density control additive used in oil muds as well as water-base muds. Calcium carbonate also is used sometimes when a relatively low mud density is required.
h. Control of Solids and Water Content: Hydro cyclones and centrifuges cannot be used economically on oil muds since a significant volume of the expensive liquid phase would

be discarded by these devices. Dilution is also quite expensive. Screening is the only economical means of solid control of oil muds.

1.1.4 Oil-base Mud

Oil-based systems were developed and introduced in the 1960s to help address several drilling problems:

1. Formation clays that react, swell, or slough after exposure to WBFs
2. Increasing downhole temperatures
3. Contaminants
4. Stuck pipe and torque and drag

Table 1.1: Diesel based mud composition

S/N	COMPONENTS	UNITS	QUANTITY
1	Diesel	Bbl	0.537
2	Organophillic Clay	lbm/bbl	6.0
3	Lime	lbm/bbl	5.0
4	Emulsifier	lbm/bbl	8.0
5	Oil wetting agent	lbm/bbl	4.0
6	Water	Bbl	0.178
7	$CaCl_2$	lbm/bbl	25.3
8	Barite	lbm/bbl	312.9
9	Density	lbm/gallon	14.0
10	OWR		75:25
11	$CaCl_2$ Brine Concentration		29%

Here are some of the desirable properties of oil-based muds, which include:

a. Shale Stability: OBM are most suited for drilling shaly formations. Since oil is the continuous phase & water is dispersed in it, this case results in non-reactive interactions with shale beds.

b. Penetration Rates: OBM usually allow for increased penetration rates.

c. Temperature: OBM can be used to drill formations where BHT (Bottom Hole Temperatures) exceed water-based mud tolerances. Sometimes up to over 1000 degrees Rankine.

d. Lubricity: OBM produces thin mud cakes, and the friction between the pipe and the well bore is minimized, thus reducing the pipe differential sticking. Especially suitable for highly deviated and horizontal wells.
e. Ability to drill low pore pressured formations is accomplished, since the mud weight can be maintained at a weight less than that of water (as low as 7.5 ppg).
f. Corrosion control: Corrosion of pipes is reduced since oil, being the external phase coats the pipe. This is due to the fact that oils are nonconductive, thermally stable, and more often, do not permit microbial growth.

Oil-based fluids (OBFs) in use today are formulated with diesel, mineral oil, or low-toxicity linear olefins and paraffins. The olefins and paraffins are often referred to as "synthetics" although some are derived from distillation of crude oil and some are chemically synthesized from smaller molecules. The electrical stability of the internal brine or water phase is monitored to help ensure that the strength of the emulsion is maintained at or near a predetermined value. The emulsion should be stable enough to incorporate additional water volume if a downhole water flow is encountered.

Barite is used to increase system density, and specially-treated organophilic bentonite is the primary viscosifier in most oil-based systems. The emulsified water phase also contributes to fluid viscosity. Organophilic lignite, asphaltic and polymeric materials are added to help control HP/HT (High pressure/High temperature) fluid loss. Oil-wetting is essential for ensuring that particulate materials remain in suspension. The surfactants used for oil-wetting also can work as thinners. Oil-based systems usually contain lime to maintain an elevated pH, resist adverse effects of hydrogen sulfide (H_2S) and carbon dioxide (CO_2) gases, and enhance emulsion stability.

Shale inhibition is one of the key benefits of using an oil-based system. The high-salinity water phase helps to prevent shales from hydrating, swelling, and sloughing into the wellbore. Most conventional oil-based mud (OBM) systems are formulated with calcium chloride brine, which appears to offer the best inhibition properties for most shales.

The ratio of the oil percentage to the water percentage in the liquid phase of an oil-based system is called its oil/water ratio. Oil-based systems generally function well with an oil/water ratio in the range from 65/35 to 95/5, but the most commonly observed range is from 70/30 to 90/10.

The discharge of whole fluid or cuttings generated with OBFs is not permitted in most offshore-drilling areas. All such drilled cuttings and waste fluids are processed, and shipped to shore for disposal. Whereas many land wells continue to be drilled with diesel-based fluids, the development of synthetic-based fluids (SBFs) in the late 1980s provided new options to offshore operators who depend on the drilling performance of oil-based systems to help hold down overall drilling costs but require more environmentally-friendly fluids. In some areas of the world such as the North Sea, even these fluids are prohibited for offshore discharge.

1.1.4.1 Advantages and Disadvantages of Oil-Base Mud

Advantages

1. Temperature Stability
2. Reduced Formation Damage
3. Enhanced Lubricity:
4. Improved Hole Cleaning
5. Better ROP (Rate of Penetration)
6. Minimized Shale Swelling and Instability
7. Improved Hole Stability
8. Effective in Contaminated Environments
9. Enhanced Drilling Fluid Properties
10. Reduction in Lost Circulation

Disadvantages

1. Expensive
2. Kick detection is reduced when using oil muds (compared to that of water-based muds) due to high gas solubility in oil muds.
3. Oil muds are costly when lost circulation occurs.
4. Greater emphasis is placed on environmental concerns when using oil muds as related to discharge of cuttings, loss of whole mud and disposal of the oil mud.
5. Special precautions should be taken to avoid skin contact which may promote allergic reactions. Inhalation of fumes from oil muds can be irritating.

6. Oil muds can be damaging to the rubber parts of the circulating system and preclude the use of special oil resistant rubber.
7. Oil muds pose potential fire hazards due to low flash points of vapors coming off the oil mud. Mineral oils and the synthetic fluids have higher flash points than diesel and crude oils. Crude oils should be "weathered" before using in oil muds.
8. Additional rig equipment and modifications are necessary to minimize the loss of oil muds.
9. Electric logging must be modified for use in oil-based muds. Oil muds are non-conductive therefore resistivity measuring logs will not work in oil muds (SP and resistivity).
10. Environmental Toxicity

1.1.5 Synthetic Mud

Synthetic-based fluid is a mud in which the base fluid is a synthetic oil. This is most often used on offshore rigs because it has the properties of an oil-based mud, but the toxicity of the fluid fumes is much less. This is important when the drilling crew works with the fluid in an enclosed space such as an offshore drilling rig. Synthetic-based fluid poses the same environmental and analysis problems as oil-based fluid. Nonaqueous, water-internal (invert) emulsion muds in which the external phase is a synthetic fluid rather than an oil. This and other more minor changes in formulations have made synthetic fluids in muds more environmentally acceptable for offshore use. Synthetic muds are popular in most offshore drilling areas, despite high initial mud costs, because of their environmental acceptance and approval to dispose of cuttings into the water. "Oil mud" should not be used to describe synthetic-base muds.

The use of synthetic-based muds offers greater waste reducing capabilities than water-based muds. They also permit drilling in areas which now require oil-based mud to combat troublesome shales. Compared to hauling oil-based mud, synthetic-based muds offer significant non-water quality advantages in the areas of air pollution, worker safety, reduction of potential spills, and reduction in landfill usage. Some key advantages of Synthetic Based Muds (SBM) over Water Based Muds (WBM) and OBMs are higher penetration rates, thermal stability, lower reservoir damage, higher lubricity, low corrosion, longer bit life and lower fluid loss (Amorin, and BroniBediako, 2020; Udoh *et al*., 2012). Though SBMs are environmentally friendly over OBMs, they are sometimes more costly. Also, not all chemically produced SBFs are biodegradable and economical like the ester (vegetable oils) SBMs.

1.1.6 Other Type of Drilling Fluid

Pneumatic (Air, Mist, Foam, Gas) Fluids

Pneumatic fluids are used in specialized applications, such as drilling through highly fractured or unconsolidated formations. They offer high penetration rates and minimal wellbore damage. However, they are not suitable for deep wells or formations with high hydrostatic pressure. Common pneumatic fluids include:

1. Dry Air: Air used as the circulating medium.
2. Mist: Air mixed with water droplets to improve hole cleaning.
3. Foam: Air mixed with foam-generating agents to increase viscosity and hole stability.

Water-Based Muds (WBMs)

WBMs are the most common type of drilling fluid, accounting for approximately 80% of all drilling operations. They are considered environmentally friendly and less expensive than OBMs. WBMs consist of a base fluid, typically freshwater or saltwater, mixed with various additives to achieve the desired properties. Common WBMs include:

1. Freshwater Muds: Used in shallow wells with stable formations.
2. Saltwater Muds: Used in deeper wells and formations with higher salinity.
3. Lime Muds: Used to control shale hydration and prevent borehole collapse.
4. Polymer Muds: Used to improve viscosity and cuttings carrying capacity.

1.1.7 Properties of Drilling Fluid to be Determined

1. Viscosity

Viscosity describes a substance’s resistance to flow. High-viscosity drilling mud is typically described as “thick,” while low-viscosity mud is characterized as “thin”. Unit of viscosity in the oil industry is centipoises (cp). In the oilfield, the following terms are used to describe drilling fluid viscosity and rheological properties.

The funnel viscosity is timed in seconds of drilling mud flowing through the Marsh Funnel Viscosity. The Marsh funnel is easy-to-use equipment that is used to quickly check viscosity of the mud. The Marsh funnel is dimensioned so that the outflow of time of one quart of freshwater (946 cc) at a temperature of 70 F ± 5 F (21 C ± 3 C) in 26 ± 0.5 seconds. For all drilling mud, especially oil-based mud, temperature has an effect on the viscosity of a base fluid. The base fluid will be less thick once the temperature increases. It means that the funnel viscosity will decrease. The funnel viscosity measures at only one rate of shear, but the temperature each time of measurement is not constant. This is the reason why the viscosity measured from the Marsh Funnel does not represent the true drilling mud viscosity.

Figure 1.2: Marsh Funnel Viscometer

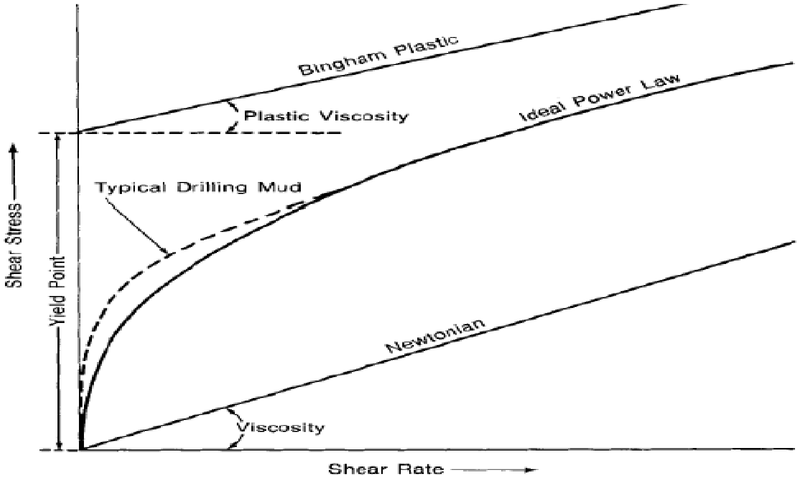


Figure 1.3: Ideal consistency curves for common flow models

2. Density

Mud weight or mud density is a weight of mud per unit volume. It is one of the most important drilling fluid properties because it controls formation pressure and it also helps wellbore stability. Mud weight is measured and reported in pounds per gallon (PPG), pounds per cubic feet (lb/ft^3), or grams per milliliter (b/ml).

Mud weight is normally measured by a conventional mud balance; however, if you have some air inside a fluid phase, reading from the conventional mud balance will give you an inaccurate number. Therefore, the most accurate method to measure the mud weight is with a pressurized mud balance. The pressurized mud balance looks like the conventional one, but it has a pressurized sample cup. When you press a mud sample into the cup, any gas in a fluid phase is compressed to a very small volume so the mud weight measurement is more accurate.

Figure 1.4: Mud balance

3. Filtration Loss

The filtration properties of drilling muds are a measure of the ability of the solid components of the muds to form a thin, low-permeability filter cake. The lower the permeability, the thinner the filter cake and the lower the volume of filtrate from muds of comparable solids concentration. This property is dependent upon the amount and physical state of the colloidal material in the mud. The filter cake building property of a mud can be measured using a FILTER PRESS.

During the test, the rate at which fluid from a mud sample is forced through a filter under specified temperature & pressure is evaluated. Also, the thickness of the solid residue deposited on the filter paper caused by the loss of fluid is measured. Note that this type of test does not accurately simulate downhole conditions in that only static filtration is being measured. In the wellbore, filtration is occurring under dynamic conditions with the mud flowing past the wall of the hole.

The loss of liquid from a mud due to filtration is controlled by the filter cake formed of the solid constituents in the drilling fluid. Two types of filtrations occur; dynamic, while circulating and static, while the mud is at rest.

Dynamic filtration reaches a constant rate when the rate of erosion of the filter cake due to circulating matches the rate of deposition of the filter cake.

Static filtration will cause the cake to grow thicker with time, which results in a decrease in loss of fluids with time. The test consists of monitoring the rate at which fluid is forced from a filter press under specific conditions of time, temperature and pressure, then measuring the thickness of the residue deposited upon the filter paper. Excessive filtration and thick filter cake build up are likely to cause the following problems:

1. Tight hole, causing excessive barrier.
2. Increased pressure, due to reduced hole diameter.
3. Differential sticking, due to an increased pipe contact in filter cake.
4. Excessive formation damage and evaluation problems with wireline logs.

Pressure also affects filtration by compressing the filter cake, reducing its permeability and therefore reducing the filtrate.

Figure 1.5: Filter press

4. pH

The degree of acidity or alkalinity of drilling mud is determined by hydrogen ion concentration. The hydrogen ion concentrations expressed in terms of pH and defined as the log of reciprocal of moles of hydrogen ion concentration per liter of solution". pH = - log (H+). Since pure water has

(H+) =(OH-) = 10-7, the pH of pure water equals 7. Thus, a neutral solution has a pH value of 7.0. The alkaline solution has pH value above 7.0 for slightly alkaline and 11.0 for the strongest alkaline.

However, acid solutions have pH from just below 7.0 for slight acid to less than 1.0 for strongest acidity. The pH measurement is used as an acid in determining the need for chemical treatment of the mud as well as indicating the presence of contaminants in mud during drilling. The pH of a mud seldom is below 7 and, in most cases, falls between 8 and 12.5 depending upon the type of mud. The pH is important because the pH affects the solubility of the organic thinners and the dispersion of clays present in the mud.

Methods of measuring pH in the laboratory:

1. The pH Paper: The pH paper strips have dyes absorbed into the paper display certain colors in certain pH ranges. It is a useful, inexpensive method to determine pH in freshwater muds. The main disadvantage is that high concentrations of salts (10,000 pm chloride) will alter the color change and cause inaccuracy.
2. The pH Meter: The pH meter is an electric device utilizing glass electrodes to measure a potential difference and indicate directly by dial reading the pH of the sample. The pH meter is the most accurate method of measuring pH.

Figure 1.6: pH meter

Figure 1.7: pH paper

5. Gel Strength

The Baroid Rheometer is also used to determine the Gel strength, in 1b/100 sq. ft, of a mud. The Gel strength is a function of the inter-particle forces. An initial 10-second gel and a 10-minute gel strength measurement give an indication of the amount of gelation that will occur after circulation ceases and the mud remains static. The more the mud gels during shutdown periods, the more pump pressure will be required to initiate circulation again.

Most drilling muds are either colloids or emulsions which behave as plastic or non-Newtonian fluids. The flow characteristics of these differ from those of Newtonian fluids (i.e., water, light oils, etc.) in that their viscosity is not constant but varies with the rate of shear, as shown in the figure above. Therefore, the viscosity of plastic fluid will depend on the rate of shear at which the measurements were taken.

Figure 1.8: Baroid Rheometer

6. Yield point

This is the measure of the electro-chemical or attractive forces in the mud under flow (dynamic) conditions. These forces depend on (1) surface properties of the mud solids, (2) volume concentrations of the solids and (3) electrical environment of the solids. The yield point of the mud reflects its ability to carry drilled cuttings out of the hole.

Measurement:

YP = 300 RPM - Plastic Viscosity

7. Sand Content

A high proportion of sand in the mud is undesirable because it can damage the mud pumps, settle in the hole about the tools when circulation is stopped and may also cause a thick filter cake on the wall of the hole. It is essential to regularly measure the sand percentage in the mud. Sand content can be determined by elutriation, settling or sieve analysis. Of the three methods, sieve analysis is the most preferred because of reliability of test & simplicity of equipment.

The sand content of the drilling fluid defines sand-sized particles larger than 74 μm in size. Excessive sand may result in the deposition of a thick filter cake on the wall of the hole, or may settle in the hole about the tool when circulation is stopped, thus, interfering with successful operation of drilling tools or setting of casings. High sand content also may cause excessive abrasion of pump parts and pipe connections. The kit consists of a special 200-mesh sieve 2½ inches in diameter, fastened inside a collar upon which a small funnel is fitted on either end. This is used with a 10 ml glass measuring tube, graduated to read from 0 to 20% the percentage sand by volume. The collar and funnel are made of polyethylene and the screen is made of brass. A 500ml wash bottle and carrying case are included. Sand content in mud is determined by the BAROID SAND CONTENT SET which consists of a 200-mesh sieve, a funnel and a glass tube calibrated in percentage by volume.

Figure 1.9: Baroid Sand Content set

1.2 Problem Statement

The environmental impact of traditional oil-based muds poses significant challenges. These muds often contain synthetic and non-biodegradable components, leading to ecological concerns and regulatory pressures.

In addressing this issue, the production and characterization of an environmentally friendly oil-based mud derived from Raphia palm seeds present an opportunity. The problem lies in the absence of a widely adopted and sustainable alternative that maintains the efficiency of oil-based muds while minimizing environmental repercussions.

1.3 Aim and Objectives

1.3.1 Aim

This research project aims to investigate the feasibility and sustainability of producing an environmentally friendly oil base mud from Raphia palm seeds (*Raphia Hookeri*). The study will involve the entire process from seed selection to mud characterization, emphasizing the potential benefits and applications in the oil and gas drilling industry.

1.3.2 Objectives

Specific objectives of this study are:

1. To prepare and characterize an API oil base mud using diesel oil.
2. To investigate the potential of Raphia palm seed oil extract for preparation of drilling fluid.
3. To prepare and characterize a standard drilling fluid with Raphia palm seed oil extract.
4. To compare the mud rheology properties of diesel oil drilling mud with that of Raphia palm seed oil-based mud.

1.4 Scope of Study

The scope of this study is limited to using only Raphia palm seeds to produce an environmentally friendly oil-base mud by investigating and optimizing the extraction process of oil from Raphia palm seeds. Exploring different techniques such as cold pressing or solvent extraction, to determine their impact on oil yield and quality. Characterization of the rheological properties of the environmentally friendly oil-based mud, including viscosity, shear rate dependency, and yield stress. Evaluation of the filtration characteristics of the mud to determine its ability to control fluid loss and maintain wellbore stability. Investigate the impact of Raphia palm seed oil on filter cake properties and permeability. Conducting a comprehensive environmental impact assessment, including biodegradability studies and toxicity assessments and comparing the environmental impact of the Raphia palm seed oil-based mud with conventional oil-based muds.

1.5 Justification

Drilling mud is in varying degrees of toxicity. It is difficult and expensive to dispose of it in an environmentally friendly manner. Protection of the environment from pollutants has become a serious task. In most countries like Nigeria, the drilling fluids industries have had numerous restrictions placed on some materials they use and the methods of their disposal. Now, at the beginning of the 1990's, the restrictions are becoming more stringent and restraints are becoming worldwide issues. In most countries today, oil-based mud may be used but not discharged in offshore or inland waters. Potential liability, latent cost, and negative publicity associated with an oil-mud spill are economic concerns. Conventional oil-based drilling muds often contain synthetic and non-biodegradable components, leading to environmental pollution and ecosystem disruption. Developing an alternative mud from Raphia palm seeds addresses these concerns and aligns with global efforts towards environmentally responsible drilling practices. The research aims to explore the economic viability of using Raphia palm seeds for mud production. If successful, this approach could not only provide an environmentally friendly solution but also contribute to cost-effective drilling practices, reducing the overall environmental and economic footprint of drilling operations. The research aligns with broader global sustainability goals, such as those outlined in the United Nations' Sustainable Development Goals (SDGs). Addressing environmental concerns in the oil and gas industry through sustainable drilling practices supports these international initiatives.

In conclusion, the proposed research on environmentally friendly oil-based mud from Raphia palm seeds is justified by its potential to address pressing environmental challenges, promote the use of renewable resources, and contribute to the paradigm shift towards sustainable and responsible drilling practices in the oil and gas industry.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Raphia Palm Seed

Raphia palm tree (*faminifera*) is the largest palm in Africa and is restricted to the tropical rainforest, the ideal ecological condition for the Raphia palm (Fabunmi et al., 2015, Ndon, 2003). The Raphia palm tree is a representative of the family of Palmae or Palmacea. There are about thirty species of the tree within the tropical and subtropical regions in the world. In Nigeria, it is represented by about five species: Raphia hookeri Mann and Wendl., Raphia sudanica Chev., Raphia vinifera Beauv., Raphia regalis Beccand Raphia farinifera (Ogbuag, 2008, Oyen and Lammens, 2002, Profizi, 1985).

Raffia palm (*Raphia hookeri*) is a medium sized tree. It has a large reddish bulbous trunk and larger feather palm leaves. It can be easily located all around the rainforest. In Nigeria, it is normally found where there are streams and rivers, although it can be found around houses. Raffia palm is an economically useful plant in Africa; the leaves are used for shelter, and the stem produces palm sap (palm wine), which is drunk as a cultural beverage. The fermented sap could be distilled into alcohol or local gin or ogogoro in Nigeria (Wallis, 2015).

Raffia oil is very similar to palm oil in chemical composition and is used for cooking, as liniment, as lubricant, for lighting and in cosmetics and could be used for making soap and margarine (Akpan and Usoh, 2004). The mesocarp of the ripe fruit yields an edible oil (Ohimain et al., 2013).

Figure 2.1: Raphia palm seeds

2.1.1 Raphia Palm Seed Oil (RPSO)

Raphia oil is very similar to palm oil in chemical composition and is used for cooking, as liniment, as lubricant, for lighting and in cosmetics and could be used for making soap and margarine (Akpan and Usoh, 2004). The mesocarp of the ripe fruit yields an edible oil.

Raphia palm seed oil (RPSO) has emerged as a promising alternative to conventional oil-based drilling fluids (OBMs) due to its biodegradability, environmental friendliness, and favorable rheological properties. Several studies have investigated the production and characterization of environmentally friendly OBMs using RPSO, demonstrating its potential as a sustainable drilling fluid component. RPSO is extracted from the seeds of the Raphia palm tree, a species abundant in tropical regions. The oil extraction process typically involves mechanical extraction, solvent extraction, or a combination of both methods. Mechanical extraction involves drying, crushing, and pressing the seeds to release the oil. Solvent extraction involves extracting the oil using solvents such as hexane or ethanol. Once extracted, RPSO is refined to remove impurities and improve its quality. The refining process may include degumming, neutralization, bleaching, and deodorization. Refined RPSO can then be used as the base oil for formulating environmentally friendly OBMs.

Several studies have evaluated the rheological properties, environmental impact, and performance of RPSO-based OBMs. These studies have demonstrated that RPSO-based OBMs exhibit favorable rheological properties comparable to conventional OBMs. They also show that RPSO-based OBMs are readily biodegradable and have a lower environmental impact than conventional OBMs.

Rheological properties are crucial for drilling fluids, as they affect their ability to transport cuttings, maintain wellbore stability, and lubricate drilling equipment. Studies have shown that RPSO-based OBMs exhibit viscosity, yield stress, and gel strength comparable to conventional OBMs. These properties can be further enhanced by adding appropriate additives. RPSO is a biodegradable oil, which means it can be broken down by microorganisms into harmless substances. This significantly reduces the environmental impact of RPSO-based OBMs compared to conventional OBMs, which are typically made from mineral oil or diesel, both of which are persistent in the environment. RPSO-based OBMs have demonstrated comparable performance to conventional OBMs in terms of wellbore stability, cuttings carrying capacity, and lubrication. Studies have shown that RPSO-based OBMs can effectively maintain wellbore stability in reactive formations and efficiently transport cuttings up the wellbore.

RPSO-based OBMs offer a promising alternative to conventional OBMs due to their biodegradability, environmental friendliness, and favorable rheological properties. Further research and development are needed to optimize the formulation and performance of RPSO-based OBMs for wider adoption in the drilling industry.

Figure 2.2: Raphia Palm Seed Oil Extract

2.1.2 Yield of Raphia Palm Seed Oil

Determining the exact yield of Raphia palm seed oil for use in oil-based mud (OBM) formulations remains an ongoing area of research, with factors like processing methods, seed varieties, and desired end product characteristics influencing the final outcome.

1. Seed Oil Content: Raphia palm seeds typically contain around 20-30% oil by weight. This implies that for every 100 kg of seeds, you could potentially extract 20-30 kg of oil, which is actually not economical.
2. Processing Methods: Different extraction methods, such as cold pressing, solvent extraction, or expeller pressing, can yield varying amounts of oil. Cold pressing generally offers the highest quality oil while potentially yielding slightly less compared to other methods.

3. Seed Variety: Several Raphia palm species exist, and their oil content can vary. Raphia farinifera, the most common species, offers the aforementioned oil content range.
4. OBM Formulation Requirements: Depending on the specific properties desired for the OBM, additional processing steps might be necessary. Refining the oil or extracting specific fractions can influence the final yield.
5. Current Research: Studies are ongoing to optimize extraction and processing methods for Raphia palm oil specifically for OBM applications. These efforts aim to improve efficiency and maximize yield while maintaining the desired oil properties.

Therefore, while it's difficult to provide a definitive yield figure for Raphia palm seed oil for OBMs at this stage, the potential seems promising. Combining the relatively high oil content of the seeds with optimized processing methods could translate to commercially viable yields. More research and field testing are necessary to establish precise oil recovery rates and determine the economic feasibility of large-scale production for OBM applications.

2.2 Raphia Palm Seed Oil Extracts: A novel frontier in oil-based mud formulation

The quest for sustainable and high-performing drilling fluids has led researchers to explore unconventional resources, and Raphia palm seed oil extracts are emerging as a promising candidate for oil-based mud (OBM) formulations. Extracted from the seeds of the Raphia farinifera palm, native to tropical Africa, these oils offer unique properties that hold potential for revolutionizing OBM performance and environmental impact.

Here's how Raphia palm seed oil extracts can contribute to OBMs:

1. Biodegradability: Unlike conventional OBMs derived from petroleum, raphia palm oil is readily biodegradable, minimizing environmental risks associated with drilling waste disposal.
2. Lubricity: The oil's natural fatty acid content imparts excellent lubricity, reducing friction between the drill string and wellbore, minimizing wear and tear on equipment and optimizing drilling efficiency.

3. Temperature stability: Raphia palm oil exhibits good thermal stability, making it suitable for use in high-temperature wells where conventional OBMs might degrade.
4. Emulsification: Specific fractions of the oil can act as natural emulsifiers, stabilizing the oil-water mixture within the OBM and preventing fluid separation.
5. Corrosion inhibition: Certain components of the oil exhibit mild corrosion inhibition properties, protecting metallic components from the harsh environment within the wellbore.

However, some challenges remain in harnessing the full potential of Raphia palm oil extracts for OBMs:

1. Viscosity control: Raphia palm oil, in its natural state, might not provide sufficient viscosity for all drilling applications. Blending with other natural or synthetic thickeners might be necessary to achieve the desired rheological properties.
2. Filtration control: The oil's low filtration rate might necessitate the use of additional filtration control agents to minimize fluid loss into formations.
3. Cost-effectiveness: Extracting and processing Raphia palm oil on a large scale for drilling applications might require optimization to make it commercially competitive with conventional OBMs.

Ongoing research and development efforts are addressing these challenges:

1. Chemical modifications: Scientists are exploring ways to modify the oil's chemical structure to enhance its viscosity, filtration control, and overall performance in OBMs.
2. Blending optimization: Identifying the optimal blends of Raphia palm oil with other natural or synthetic additives to achieve the desired OBM properties for specific drilling needs.
3. Sustainable sourcing: Establishing sustainable practices for cultivating and harvesting Raphia palms to ensure long-term availability and minimize environmental impact.

The potential benefits of Raphia palm seed oil extracts for OBMs are undeniable. Its biodegradability, lubricity, and temperature stability offer a glimpse into a more sustainable and efficient future for drilling operations. With continued research and development, raphia palm oil extracts could pave the way for a new generation of environmentally friendly and high-performing OBMs, revolutionizing the drilling industry. Remember, the use of raphia palm seed oil extracts in OBMs are still in its early stages of development. Further research and testing are necessary to fully assess its technical and economic viability for broad-scale drilling applications. However, the potential of this renewable resource is promising, and its continued exploration holds exciting possibilities for the future of sustainable drilling.

2.3 Composition of Raphia palm seed oil base mud (RPSO-based OBM)

However, we can break down the potential components of a raphia palm seed oil base mud based on what we know about the oil and the typical components of an OBM:

1. Base Oil:

Raphia palm seed oil would be the primary component, constituting around 50-70% of the mud volume. Its naturally occurring fatty acids like oleic acid and linoleic acid provide lubricity and some viscosity.

Figure 2.3: Sun-dried Raphia Palm seeds

2. Viscosity Modifiers:

Natural thickeners: Depending on the desired rheology, natural thickeners like cellulose fibers or xanthan gum might be added to enhance mud viscosity and improve hole cleaning.

3. Synthetic polymers:

In some cases, synthetic polymers like polyacrylamide could be used for precise viscosity control, especially at high temperatures.

Figure 2.4: Synthetic polymer (Polyacrylamide)

4. Emulsifiers:

Specific fractions of raphia palm oil: Certain fractions extracted from the oil itself can act as natural emulsifiers, stabilizing the oil-water mixture within the mud.

Additional emulsifiers: Depending on the desired emulsification properties, surfactants or other commercial emulsifiers might be included.

5. Filtration Control Agents:

Calcium carbonate: Ground calcium carbonate can act as a mild filtration control agent by plugging small pores and fissures in formations.

Diatomaceous earth: This naturally occurring silica powder can form a low-permeability layer on the wellbore wall, minimizing fluid loss.

6. Corrosion inhibitors:

These protect metallic components from the harsh environment within the wellbore.

7. Lost circulation materials (LCMs):

In case of formation fractures, materials like groundnut shells or walnut shells could be added to temporarily plug the gaps and maintain wellbore pressure.

2.4 Emulsion

An emulsion is a mixture of two or more liquids that are normally immiscible, such as oil and water. Emulsions can be either temporary or stable, depending on the nature of the mixture and the presence of certain agents called emulsifiers.

Two liquids can form different types of emulsions. As an example, oil and water can form, first, an oil-in-water emulsion, in which the oil is the dispersed phase, and water is the continuous phase. Second, they can form a water-in-oil emulsion, in which water is the dispersed phase and oil is the continuous phase. Multiple emulsions are also possible, including a "water-in-oil-in-water" emulsion and an "oil-in-water-in-oil" emulsion. Another type of emulsion, known as a Pickering emulsion, is stabilized by solid particles rather than a liquid emulsifier.

Emulsifiers, also known as surfactants, are chemicals that are added to emulsions to improve their stability. These chemicals work by reducing the surface tension between the oil and water phases, which helps to prevent the droplets from coalescing and separating. Wetting agents are also commonly used in emulsions to improve the wetting and spreading characteristics of the mixture. These chemicals help to distribute the droplets evenly throughout the emulsion, which improves its stability and overall performance.

Emulsification is the process by which the dispersed phase is broken up into small droplets. Normally a coarse premix is created by rapid mixing of the ingredients. This is sufficient to break up the dispersed phase into large droplets, and allow adsorption of the emulsifiers prior to final emulsification. The stability of an emulsion is typically measured using various tests and techniques, such as sedimentation tests, stability measurements, and droplet size analysis. These tests help to determine the effectiveness of the emulsifiers and other chemicals used in the emulsion, and can provide valuable information for optimizing the emulsion formulation and performance.

In the world of oil and gas, emulsification refers to the formation of a mixture where two immiscible liquids, typically oil and water, coexist in the same space. These liquids don't naturally form a stable blend and tend to separate. Drilling fluids are used to lubricate and cool the drill bit and to carry cuttings out of the well. These fluids typically contain emulsifiers and oil wetting agents, which help to improve the stability and performance of the fluid.

However, under specific conditions, emulsifiers and shear forces can bring them together, creating a stable emulsion.

2.4.1 Types of Emulsions:

1. Oil-in-water (O/W): In this type, oil droplets are dispersed throughout a continuous water phase. This is the more common type encountered in oil and gas production.
2. Water-in-oil (W/O): Here, water droplets are dispersed in a continuous oil phase. These are less frequent but can occur in certain situations.

2.4.2 Types of Emulsifiers:

a. Primary emulsifiers are typically made using chemicals such as fatty acids, fatty alcohols, and glycerol, which are derived from natural sources such as vegetable oils and animal fats. These chemicals are used to create the emulsifying agents that are added to emulsions to improve their stability.

b. Secondary emulsifiers are often made using chemicals such as polymers and copolymers, which are derived from synthetic sources such as petrochemicals. These chemicals are added to emulsions in order to enhance their stability and prevent the formation of larger droplets. Secondary emulsifiers are sometimes added to emulsions in order to enhance their stability and prevent the formation of larger droplets. These chemicals can help to maintain the small droplet size and prevent the emulsion from breaking down over time.

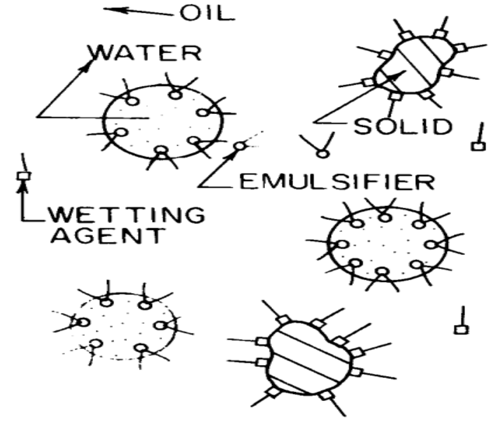


Figure 2.5: Schematic of an oil base mud from Applied Drilling Engineering **(**Bourgoyne,1986)

2.4.3 Emulsifying Agents

Emulsifying agents, also known as emulsifiers, are substances that allow two immiscible liquids to mix and form a stable mixture called an emulsion. These immiscible liquids, such as oil and water, naturally repel each other and would normally separate if left alone.

2.4.3.1 Types of emulsifying agents used in oil and gas:

1. Naturally occurring emulsifiers: These are present in crude oil itself, such as asphaltenes. They act as natural stabilizers for water-in-oil emulsions, but their effectiveness can vary depending on the specific crude oil composition.

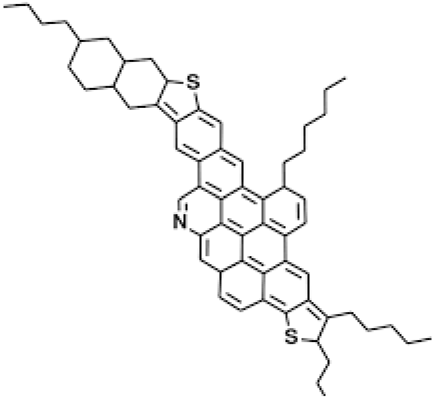


Figure 2.6: Asphaltenes molecule

2. Added emulsifiers: These are synthetic chemicals specifically formulated to enhance emulsification or demulsification: Surfactants (surface-active agents). The most common type of added emulsifier, they have a unique structure with one end attracted to water (hydrophilic) and the other attracted to oil (hydrophobic). This allows them to concentrate at the oil-water interface, reducing interfacial tension and promoting the formation of stable emulsions.
3. Demulsifiers: These are specialized surfactants designed to break down existing emulsions. They work by disrupting the stabilizing effect of natural or added emulsifiers at the oil-water interface, allowing the two liquids to separate.

2.4.3.2 Types of surfactants:

a. Anionic surfactants: With a negatively charged head group, they are effective in high pH (alkaline) environments and commonly used for oil-in-water emulsions.

b. Cationic surfactants: Having a positively charged head group, they are suitable for water-in-oil emulsions but less frequently used in oil and gas due to instability in high pH environments.

c. Non-ionic surfactants: Lacking charged groups, they can be used in various pH conditions and are often used alone or combined with other types for desired properties.

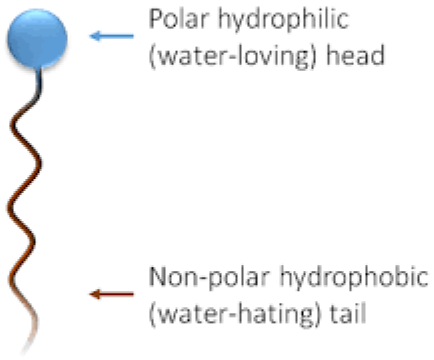


Figure 2.7: Surfactant molecule

2.4.3.3 Functions of Emulsifiers:

In the oil and gas industry, emulsifiers play a critical role in managing emulsions, mixtures of two immiscible liquids, typically oil and water. These emulsions can form naturally during the extraction process, and emulsifiers help manipulate them in two key ways:

1. Facilitating Emulsification (Stabilization)

This is the more common function of emulsifiers. They promote the formation and stability of emulsions, particularly desirable water-in-oil (W/O) emulsions. Here's how they achieve this:

a. Reducing interfacial tension: Emulsifiers have a special molecular structure. One end is attracted to oil (hydrophobic), while the other end is attracted to water (hydrophilic). This allows them to concentrate at the interface between oil and water droplets. By accumulating there, they lower the interfacial tension, which is the resistance to the expansion of the interface. This reduced tension prevents the droplets from merging (coalescing) and forming larger droplets, thus stabilizing the emulsion.
b. Forming a protective layer: Emulsifier molecules adsorb, or cling, to the surface of the water droplets. This forms a protective barrier that hinders the droplets from directly interacting with the surrounding oil, further contributing to the overall stability of the emulsion.

2. Breaking Down Emulsions (Demulsification)

While less common than stabilization, some emulsifiers are specifically designed to break down existing emulsions. These are called demulsifiers and work by:

a. Disrupting the stabilizing effect: Demulsifiers are formulated to disrupt the protective layer formed by natural or added emulsifiers around the water droplets. This allows the water droplets to overcome the interfacial tension and coalesce, separating from the oil phase.
b. Promoting separation: By destabilizing the emulsion, demulsifiers make it easier to separate the oil and water using various techniques like gravity separation or centrifugation.

2.4.4 Challenges of Emulsions:

While emulsions can exist naturally in reservoirs, their presence in produced fluids creates several challenges:

1. Difficulties in separation: Separating the oil from the water becomes a complex and energy-intensive process, impacting production efficiency and profitability.

2. Corrosion: Water in emulsions can accelerate corrosion in pipelines and equipment, leading to costly maintenance and potential safety hazards.
3. Transportation issues: Emulsions can be difficult and costly to transport due to their increased viscosity and potential for pipeline blockages.

2.4.5 Addressing the Issue:

To overcome these challenges, various dewatering or demulsification techniques are employed:

1. Chemical demulsifiers: Specific chemicals can be added to break down the emulsion by disrupting the action of the natural or added emulsifiers.
2. Heat treatment: Applying heat can reduce the viscosity of the emulsion and facilitate separation.
3. Electrostatic separation: This method utilizes an electric field to separate the oil and water based on their different electrical properties.

Overall, emulsifiers are important chemicals used in various industries, including the oil and gas industry, to improve the stability and performance of emulsions. These chemicals play a crucial role in enabling the effective transport and storage of feedstocks, and are essential for maintaining the efficiency and effectiveness of various processes and operations.

2.5 Surfactant

Surfactants are essential chemicals and play a significant role in the upstream petroleum industry. Their interfacial properties and strong emulsification capabilities make them suitable for many operations involving surface and interfaces. Apart from that, surfactants are important chemicals and are widely used for other processes as well. They have an important role in demulsification, fracturing, drilling, cement slurries, acidization, transportation, corrosion inhibition.

They are surface active agents which are polymeric molecules that lower the IFT between the liquid surfactant solution and the residual oil. Surfactants adsorb on a surface or fluid/fluid interface when present at low concentrations. The most common structural form for surfactants is where they contain a nonpolar part, a hydrocarbon ‘tail’, and a polar or ionic part.

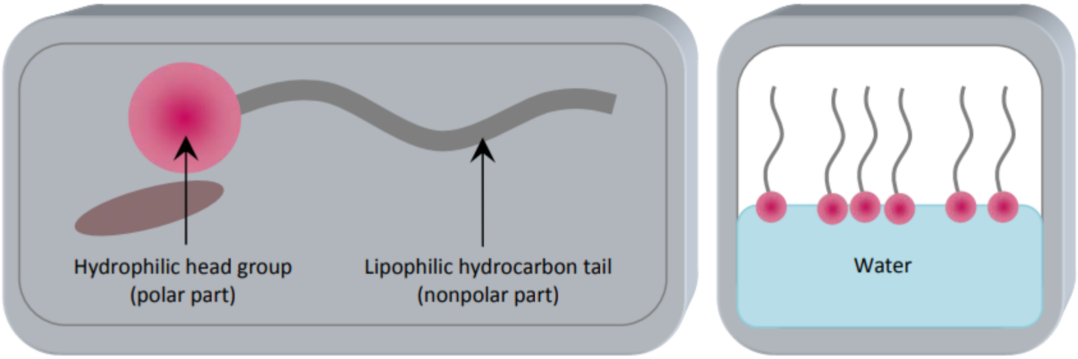
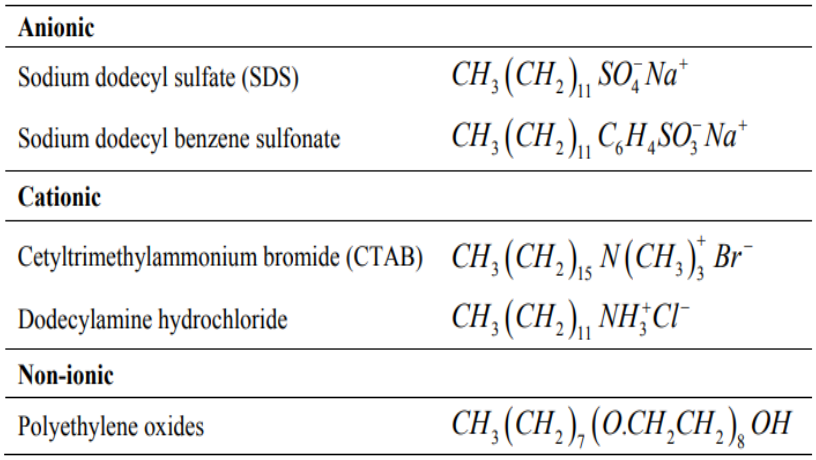


Figure 2.8: Surfactant molecule and surfactant orientation in water (Green and Willhite, 1998)

It is the balance between the hydrophilic and hydrophobic parts of the surfactant that generates the characteristics of the surface-active agent. Surfactant molecules are amphiphilic, as they have both hydrophilic and hydrophobic moieties. Amphiphiles adsorb effectively to interfaces and typically contribute to significant reductions of the interfacial energy, [Pashley and Karaman, 2004, p. 62]. The primary surfactant is directly involved in the microemulsion formation with regards to the EOR surfactant flooding process. The co-surfactant, if any, promotes or improves the activities of the primary surfactant, by e.g., changing the surface energy or the viscosity of the liquids. Due to chromatographic separation of surfactant, co-surfactant and any other components, throughout the reservoir, it can be problematic to create a multicomponent surfactant system capable of maintaining optimal properties throughout the flooding process. The predominant disadvantage of separation is that the control of the system deteriorates in the reservoir and therefore it should be avoided if possible. As the co-surfactants prevent gel formation and reduce the equilibration time, they are hard to eliminate from the surfactant systems used for flooding. Oil reservoirs have different characteristics and therefore the structure of added surfactant must be tailored to meet the reservoir conditions to achieve a low IFT. For example, the temperature, pressure and rock vary significantly from one reservoir to another.

2.5.1 Classification of Surfactants

Surfactants are frequently classified on the basis of the ionic nature of the head group, as anionic, cationic, nonionic or zwitterionic. Each type possesses certain characteristics depending on how the surfactant molecules ionize in aqueous solutions. In figure 2.9 a few commonly used surfactants are shown.



Anionic	
Sodium dodecyl sulfate (SDS)	$CH_3(CH_2)_{11}SO_4^-Na^+$
Sodium dodecyl benzene sulfonate	$CH_3(CH_2)_{11}C_6H_4SO_3^-Na^+$
Cationic	
Cetyltrimethylammonium bromide (CTAB)	$CH_3(CH_2)_{15}N(CH_3)_3^+Br^-$
Dodecylamine hydrochloride	$CH_3(CH_2)_{11}NH_3^+Cl^-$
Non-ionic	
Polyethylene oxides	$CH_3(CH_2)_7(O.CH_2CH_2)_8OH$

Figure 2.9: List of common surfactant molecules with different types of charge (Pashley & Karaman, 2004)

The demands on surfactants are numerous and it is a great challenge to distinguish which mechanisms are most dominant. Process conditions, such as high temperature and high pressure are often the reality in reservoir environments.

2.5.1.1 Use of Anionic Surfactants

Anionic surfactants are negatively charged. They are commonly used for various industrial applications, such as detergents (alkyl benzene sulfonates), soaps (fatty acids), foaming agents (lauryl sulfate), and wetting agents (di-alkyl sulfosuccinate). Anionic surfactants are also the most commonly used in EOR. They display good surfactant properties, such as lowering the IFT, their ability to create self-assembled structures, are relatively stable, exhibit relatively low adsorption on reservoir rock and can be manufactured economically [Green & Willhite, 1998, p. 241]. Anionic surfactants dissociate in water to form an amphiphilic anion (negatively charged) and a cation (positively charged), which would typically be an alkaline metal such as sodium (Na+) or potassium (K+).

Barnes et al. (2008) investigate families of anionic surfactants, internal olefin sulfonates, (IOS), for use in surfactant flooding at high temperatures, (up to 150 °C), and with varying optimal salinities from 1 % to 13 % depending on the carbon number range. The IOS surfactants show little sensitivity to temperature, which could be an advantage for reservoirs with temperature gradients. Overall, the IOS surfactants exhibit promise over a range of reservoir conditions covering moderate to high temperatures and from low to high salinity conditions.

2.5.1.2 Use of Nonionic surfactants

Nonionic surfactants have no charged head group. They are also identified for use in EOR, [Gupta and Mohanty, 2007], mainly as co-surfactants to promote the surfactant process. Their hydrophilic group is of a non-dissociating type, not ionizing in aqueous solutions. Examples of nonionic surfactants include alcohols, phenols, ethers, esters or amides. Curbelo et al. (2007) studied nonionic surfactants with different degrees of ethoxylation to investigate the correlation with the adsorption of surfactant in porous media (sandstone).

Critical Micelle Concentration (CMC) is reached at a higher surfactant concentration for (B), with ethoxylation degree of 15.0, compared to (A), with ethoxylation degree at 9.5. With higher ethoxylation degree follows that the surfactant has a larger polar chain and consequently higher solubility towards the aqueous phase. Thus, higher concentration of surfactant is required to assure formation of micelles. Curbelo et al. (2007) concluded that the adsorption to the sandstone core is higher in the case of the lower degree of ethoxylation, situation (A), which should be avoided in EOR surfactant flooding.

2.5.1.3 Use of Cationic Surfactants

Cationic surfactants have a positively charged head group. Cationic surfactants dissociate in water, forming an amphiphilic cation and anion, typically a halide (Br-, Cl- etc.). During the synthesis to produce cationic surfactants, they undergo a high-pressure hydrogenation reaction, which is in general more expensive compared to anionic surfactants. As direct consequence cationic surfactants are not as widely used as anionic and nonionic surfactants. It is, however, reported that cationic surfactants can be used to improve the spontaneous imbibition rate of water into preferentially oil-wet carbonate. Water containing surfactants of the type alkyl trimethylammonium bromide or chloride was injected [Standnes & Austad, 2002]. The cationic surfactants are most likely dissolved in the oil phase as aggregates between the surfactant and the carboxylates, under creation of ion pairs. In this way the surface becomes more water-wet, thus the aqueous phase can better imbibe by capillary forces.

2.5.2 Function of Surfactants in Drilling Mud

Surfactants are a type of additive used in drilling muds to perform several important functions:

1. **Wetting Agent:** Surfactants reduce the surface tension of water, allowing it to spread more easily over drilled cuttings and formation rock. This helps to improve hole cleaning and prevent formation damage.
2. **Emulsification:** In oil-based muds, surfactants help to stabilize emulsions, which are mixtures of oil and water. This is crucial for maintaining the stability of the mud and preventing the separation of the oil and water phases.
3. **Dispersion:** Surfactants can help to disperse solids in drilling muds, such as clays and weighting materials. This prevents these solids from settling out and causing problems with mud rheology (flow properties).
4. **Fluid Loss Control:** Some surfactants can help to reduce fluid loss from drilling muds to the formation. This is important for preventing formation damage and maintaining wellbore stability.

2.6 Previous Work Done on Drilling Fluid

A very noticeable amount of work has been done on using Raphia Palm seed-based oil in production and characterization of an environmentally friendly oil base mud and its application in drilling engineering. These works all have a common aim of finding greener, low-cost yet efficient techniques and methods to be used in production and characterization of an environmentally friendly oil base mud as conventional diesel oil base mud is expensive and not environmentally friendly.

According to (Rodriguez and Katz, 2021), they carried out research on the effect of oil-based drilling mud (OBM) on the assessment of hydrocarbon charge potential. Foundational to source rock assessment in exploration is an understanding of organic enrichment and hydrocarbon generation potential. In this study, an attempt was made to characterize this extraction process on a suite of simulated cuttings samples that were contaminated and extracted prior to analysis to validate the assumptions presented in the literature. Results from these experiments show significant differences between values measured on the original and oil-based drilling fluid contaminated samples before and after extraction. After contamination and before extraction, measured the total organic carbon (TOC) and Rock-Eval parameters indicate that the oil-based drilling fluids appear to be acting as an aggressive solvent, altering the apparent source rock potential. Contamination by oil-based drilling fluid appears to reduce the total organic carbon content (TOC) and the residual generation (S2) potential, while increasing the free hydrocarbon content. This may be a result of the presence of solid bitumen or bituminite in the original sample, which are acted on by the oil-based drilling fluid. Following extraction of the contaminated samples, the TOC and residual generation potential underwent further reduction and the free hydrocarbons were nearly eliminated. It was also observed that uncontaminated samples, when extracted, experienced an increase in TOC and the S3 (CO2) peak.

(Adewale and Ogunrinde, 2019) compared the eco-toxicological properties of conventional base oil of diesel and vegetable oils derived from palm oil and groundnut oil. A drilling mud was formulated with both palm oil and groundnut oil and standard additives were added. Complete mud check was conducted on the mud in order to determine the rheological properties. Toxicity of diesel, palm oil and groundnut oil were compared by exposing corns planted on humus soil beds prepared with palm. Also, groundnut oil-based mud with an oil/water ratio of 90/10 was formulated successfully. Palm oil-based mud having 90/10 oil/water ratio and groundnut oil-based mud exhibited a 20% and 12% average rate of growth without losing all its greenness. Palm oil-based mud formulated with different oil/water ratios congealed during the course of formulation. They exhibited high viscosity and progressive gel characteristics. In conclusion, oil-based mud developed using Palm oil and groundnut oil should be encouraged since its highly biodegradable, they have better eco-toxicological properties and the cost of treatment of the cuttings are lower compared to Oil-based drilling mud formulated with diesel, mineral and conventional synthetic oil.

A novel oil-in-water (O/W) emulsion drilling fluid formulated with a methyl ester extract from Indian mango seed oil was evaluated by (Kumar *et al.*, 2019). The effect of the weight percent of the different constituents of the emulsion/suspension; including the oil phase, the bentonite, and the polyanionic cellulose polymer on the rheology and the fluid loss are examined. The methyl ester oil phase/mud system displayed superior physical, chemical, rheological and filtration properties relative to the diesel and the mango seed oil. Eco toxicity of the methyl ester and the diesel (O/W) emulsion mud systems were assessed using the acute lethal concentration test. The Indian mango methyl ester (O/W) emulsion mud displayed much less impact on fish population. Flow characteristics collected from the flow model at 85°C suggest excellent shear thinning behavior of the Indian mango methyl ester (IMME) (O/W) emulsion mud.

Drilling muds capable of drilling shale sensitive formations with negligible environmental effects are in great demand. (Kesarwani, Saxena and Sharma, 2020) worked on "Novel Jatropha Oil Based Emulsion Drilling Mud Outperforms Conventional Drilling Mud: A comparative study". The work studies a 40% water in Jatropha oil invert emulsion illustrating a comparative study of the Jatropha oil invert emulsion, and conventional diesel oil-based mud (OBM). Jatropha oil is environmentally, chemically, and economically a better substitute for Diesel. The GC-MS (Gas Chromatography-Mass Spectrometry) confirms the composition of Jatropha oil and diesel oil are almost similar having a similar carbon number range. The effect of temperature, concentration of oil, and various additives on the rheology of Jatropha oil invert emulsion mud was investigated. The reduction in viscosity of Jatropha oil inverts emulsion mud after hot rolling at 16h and 105°C was around 25% as compared to 56% with Diesel oil invert emulsion mud. The Jatropha oil invert emulsion mud was thermally more stable than Diesel oil invert emulsion mud. A drastic reduction in the fluid loss from 13 to 3.4 ml was observed while LPLT (low temperature low pressure) filter press test for Jatropha oil invert emulsion mud along with the mud cake thickness of only 0.793 mm (1/32 inch). The shale recovery test confirms the usage of Jatropha oil mud in dispersive shales.

(Wajheeuddin and Hossain, 2018) developed an Environmentally-Friendly Water-Based Mud System Using Natural Materials. This article proposes three naturally occurring materials—date seeds, powdered grass, and grass ash as possible additives in the drilling mud system. Sieve analysis and laser particle size analysis are conducted to study the particle size of the three materials. Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX) (SEM–EDX) analysis is performed to know the elemental composition of the proposed additives. Experimental tests on rheology and filtration are conducted at room temperature on the proposed materials to determine the applicability of these natural substitutes as drilling fluid additives.

Results show that date seeds, grass, and grass ash can be used as a rheology modifier, and a filtration control agent to formulate the water-based mud system.

(Oseh *et al.*, 2019) investigated almond seed oil as potential biodiesel-based drilling mud, with increasing strict environmental laws, there is a need for operators to design benign oil-based muds (OBMs). In this study, oil extracted from non-edible sweet almond seed (SASO) was used as the continuous phase to formulate biodiesel-based drilling mud (BBDM). Different properties of the BBDM including the economic viability were evaluated and compared with those of the diesel OBM to determine the applicability of these properties for drilling fluids and their level of toxicity to the environment. The results indicate that the rheology, filtration properties, electrical stability, thermal stability and shale swelling inhibition performance of the BBDM are comparable with those of the diesel OBM. The biodiesel has a significantly higher flash point of 169 °C than the diesel with 78°C, demonstrating that it can supply better fire safety than the diesel. The data of the toxicity test indicate SASO to be safer and less harmful compared to diesel 2 types used. After the 28-day period of biodegradation tests, the BBDM and the diesel OBM showed 83% and 25.2% aerobic biodegradation with Penicillium sp., respectively. The low branching degree and absence of aromatic compounds in the BBDM contributes to its higher biodegradation. The economic evaluation of the BBDM indicates low cost of formulation and waste management. The general outcome of the tests illustrates that SASO has the potential of being one of the technically and environmentally feasible substitutes for the diesel OBM.

A machine Learning Model for Monitoring Rheological Properties of Synthetic Oil-Based Mud was carried out by (Alsabaa *et al.*, 2022). The current study introduces a machine learning application toward predicting the rheology of synthetic oil-based mud (flat rheology type) for the full automation system of monitoring the mud rheological properties. Four models are developed, for the first time, to determine the rheological characteristics of a flat rheology synthetic oil-based system using artificial neural networks. The developed models are capable of predicting the plastic and apparent viscosities, yield point, and flow behavior index from only the mud density and Marsh funnel as model inputs. The proposed models were trained and optimized from a real field dataset (369 measurements) with further testing the models using an unseen dataset of 153 data points. The predicted rheological properties achieved a high degree of accuracy versus the actual measurements and showed a coefficient of correlation range from 0.91 to 0.97 with an average absolute percentage error of less than 9.66% during the training and testing phases. Besides, machine learning-based correlations are proposed for estimating the rheological properties on the rig site without running the machine learning system for easy field applications.

(Onuh *et al.,* 2020) researched on the rheological behavior of a pseudo-oil-based mud formulated with Hura crepitans plant oil as base fluid. This research work involves using a commercial synthetic oil from the oil industry and *Hura crepitans* oil. These oil samples were used as a base fluid in preparing the mud from which the rheological properties were analyzed. Chemical oil extraction method using Soxhlet apparatus was used to extract the oil from *H. crepitans* seeds; it was then distilled to remove the solvent. The mud samples were formulated with 7 and 9 g concentrations of the viscosifier, and properties were measured at 113 and 158 °F. It was then aged for 16 h at 240°F, and mud properties were measured before and after hot rolling for comparison. Different rheological models were used to describe the experimental data. The physical properties of the synthetic oil and *H. crepitans* oil reveal a flash point of 213.8 and 399.2°F, fire point of 226.4 and 500°F, viscosity index of 297 and 207, specific gravity/density of 805 and 907, respectively. The mud properties of the synthetic oil-based mud had a better emulsion stability, lower plastic viscosity, higher yield point values, and lower gel strength than the *H. crepitans* oil-based mud. The rheological properties of synthetic and *H. crepitans* oil-based mud increase and decrease, respectively, after hot rolling.

Synthetic-Based Mud Systems Offer Environmental Benefits Over Traditional Mud Systems was a research work reviewed by (E. Candler *et al.,* 2018). This paper addresses critical issues concerning the regulation of synthetic-based fluids in Outer Continental Shelf waters. Synthetic-based muds were not envisioned when discharge criteria were formulated a decade ago. It is critical that the U.S. The Environmental Protection Agency investigated this new category, since the original permit language and discharge criteria may inhibit the utilization of this new technology and prevent the realization of volume reduction that can be achieved using synthetic-based muds. To date, the EPA has not addressed the use of inhibitive mud systems as a control technology for reducing the quantity of pollutants. Drilling through shale formations is an integral part of drilling wells in the Gulf of Mexico. Solving problems associated with these formations can benefit the EPA, industry, and the environment. The use of synthetic-based muds offers greater waste reducing capabilities than water-based muds. They also permit drilling in areas which now require oil-based mud to combat troublesome shales. Compared to hauling oil-based mud, synthetic-based muds offer significant non-water quality advantages in the areas of air pollution, worker safety, reduction of potential spills, and reduction in landfill usage.

(Dankwa *et al.,* 2018) Investigated the Potential Use of Waste Vegetable Oils to Produce Synthetic Base Fluids for Drilling Mud Formulation. In this research, waste or used oils from restaurants and food joints have been tested for their potential use as synthetic based fluids through rheological analysis. Physicochemical properties of the waste vegetable oil were determined; density: 0.88g/cm^3, acid value: 64.8mg KOH/g of oil and Free Fatty Acid (FFA) content: 32.4%. The optimum conditioning of biodiesel production from waste oil was in a two-step catalyzed process. In the first step, sulfuric acid was used as a catalyst for esterification reaction to reduce acid value of the oil below 3mg KOH/g which was with different dosages. The next step was the base catalyzed transesterification process which converted the pre-treated oil into biodiesel and glycerol in the presence of methanol and NaOH at varied reaction conditions. Density of 9.1 lb/gal and rheological properties:

S/N	Rheological Properties	Test 1	Test 2	Test 3
1	Yield Point	17 $lb/100ft^2$	2 $lb/100ft^2$	2 $lb/100ft^2$
2	Plastic Viscosity	17cP	8cP	6cP
3	Gel Strength @10Ỹ	3 $lb/100ft^2$	2 $lb/100ft^2$	2 $lb/100ft^2$
4	Gel Strength 10’’	5 $lb/100ft^2$	4 $lb/100ft^2$	2 $lb/100ft^2$

were all determined at temperatures of 80°F, 120°F and 160°F respectively. The formulated mud showed that biodiesel is a promising synthetic based fluid and has most of its rheological properties meeting the API standard. Though the initial cost of conditioning biodiesel will be quite high, it can be offset by its disposal cost as compared to that of diesel.

CHAPTER THREE

3.0 RESEARCH METHODOLOGY AND MATERIALS

This chapter aims to show the apparatuses, procedures and methods of extraction, production and the characterization of synthetic oil base mud from Raphia Palm seeds.

3.1 Equipment and Materials

3.1.1 Equipment

1. Weighing balance
2. Beakers
3. Spatula
4. Conical flasks
5. Soxhlet apparatus
6. Pycnometer
7. Magnetic heating stirrer
8. Heating Mantle
9. Retort stand
10. Thermometer
11. pH meter
12. Separating funnel
13. Rheometer
14. Filter paper

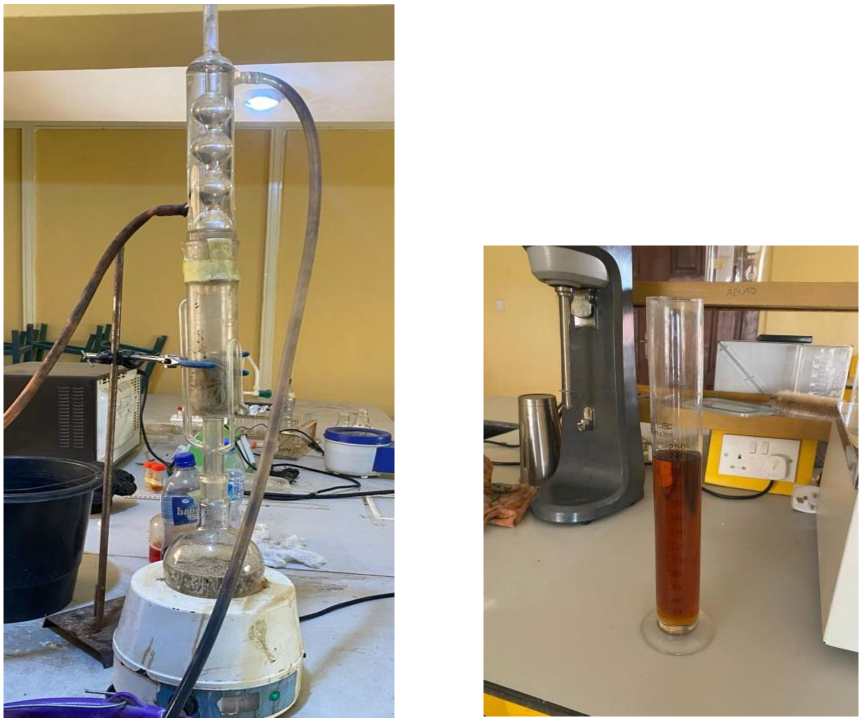


Figure 3.1: Soxhlet apparatus held by the retort stand and a measuring cylinder containing diesel

3.1.2 Materials

1. Raphia Palm seeds
2. n-hexane
3. Barite
4. Bentonite
5. CMC (Sodium Carboxyl Methyl Cellulose)
6. Organophilic clays
7. Diesel oil
8. NaOH
9. Pac R & Pac L
10. Primary & Secondary Emulsifier
11. Distilled water

Figure 3.2: Diesel and chemical additives needed for mud formulation

3.2 Research Approach

This research project involves the extraction of oil from Raphia Palm seeds, production of an environmentally friendly oil base mud from the extracted oil and comparison of the produced synthetic oil base mud with a diesel oil base mud.

3.2.1 Raphia Palm Seed Oil Extraction Process

The Raphia palm seed was obtained from a local farm in Ado-Ekiti, Ekiti state, Nigeria.

These steps were taken accordingly.

1. The Raphia palm seed was washed with water and kept under the sun for about 24 hours to dry till it was crushable as shown in figure 3.3

Figure 3. 3: Pulverized Raphia palm seed

2. The total weight of the pulverized seeds was measured using the weighing balance.

3. Some of the pulverized seeds were put in a thimble which was inserted into the extraction chamber of the Soxhlet apparatus.
4. A round bottom flask filled with 350ml of n-hexane (which was used as a solvent) and a condenser was properly fixed to the apparatus.
5. Cool water flowing constantly through the condenser was used to condense the evaporating solvent back into the Soxhlet apparatus at a temperature of 700°C.
6. The magnetic heating stirrer was set to 400°C and the extraction started.
7. When the n-hexane began boiling, the vapor was passed through the condenser, which condensed it to liquid and then dropped into the sample until the thimble was filled
8. As the liquid dropped into the sample, it extracted the oil and when the thimble was filled up the oil extracted together with the n-hexane was emptied into a round bottom flask.
9. After about 2 hours of each run, the mixture of n-hexane and the extracted oil was separated by gently heating the mixture to evaporate the n-hexane.
10. The total weight of the extracted oil was taken and the oil yield was calculated using equation (3.1)

$$\%\ \text{oil yield} = \frac{Weight\ of sample\ before\ extraction - Weight\ of\ sample\ after\ extraction}{weight\ of\ sample\ before\ extraction} \times 100\% \qquad (3.1)$$

3.2.2 Characterization of the Raphia Palm seed oil

This involves the various processes that were used to attain the physicochemical properties of the oil. These tests were density, specific gravity, viscosity, pH, yield point, acid value, free fatty acid, cloud point, pour point, saponification tests.

3.2.2.1 Density Test

1. The weight of an empty pycnometer with a stopper was taken.
2. The empty pycnometer was filled with distilled water and the weight was taken. The volume of water that is filling the pycnometer at the stopper is:

$$Volume\ of\ water = \frac{a}{b} \qquad (3.2)$$

Where a= mass of water which is experimentally used to determined weight of water, (weight of pycnometer filled with distilled water- weight of empty pycnometer) in grams and b = density of water in g/cm^3.

Figure 3.4: Pycnometer

3. The procedure was repeated for the oil of unknown density and its weight ‘m’ was determined (measured weight - weight of empty pycnometer).
4. The density was calculated with the volume of water from equation (3.2)

$$\text{Density of oil } (\rho) = \frac{mass\ of\ oil}{volume\ of\ reference\ fluid(water)} \tag{3.3}$$

5. The specific gravity was also calculated

3.2.2.2 Specific Gravity

The specific gravity of a substance can either be calculated or measured:

1. If the density of a substance is known, to calculate the specific gravity of a substance simply divide the density of the substance by the density of water or air.

$$\text{Specific gravity of oil } (\gamma) = \frac{density\ of\ oil\ extracted}{density\ of\ water} \tag{3.4}$$

The density of water is $1000 kg/m^3$ or 1 g/cm^3

2. A hydrometer is a popular way to measure the specific gravity of a liquid. A hydrometer is a bulb attached to a stalk with measurement markers that floats in liquid. By measuring how far the stalk is submerged in the target liquid compared to a reference liquid, the relative density of the two can be determined.

Figure 3.5: Hydrometer

3.2.2.3 Viscosity Test

Viscosity describes a fluid's internal resistance to flow and may be thought of as a measure of fluid friction. All real fluids (except superfluid) have some resistance to stress and therefore are viscous, but a fluid which has no resistance to shear stress is known as an ideal fluid or inviscid fluid. The study of flowing matter is known as rheology, which includes viscosity and related concepts (Kiselev, Vorozhtsov and Fomin, 2017). These were the steps:

1. The Brookfield rheometer was turned on and a spindle with a spindle factor of 0.01 was attached to it due to the amount of oil used.
2. An RPM of 250 was selected and the rheometer was auto-zeroed.
3. The oil was poured in a 50ml beaker and was heated to about 50°C. It was attached to the rheometer and the spindle was put into it.
4. The rotation started and the dynamic viscosity was taken when the temperature of the oil was at 40°C

Figure 3.6: Brookfield Rheometer

3.2.2.4 pH Test

pH shows how acidic or basic an aqueous solution is. A pH of less than 7 indicates that an aqueous solution is acidic, whereas a pH of greater than 7 indicates that it is basic. A pH of 7 is regarded as neutral. The oil was placed in a beaker and the pH probe was placed in it until the pH meter displayed "ready" with a value.

Figure 3.7: pH meter

3.2.2.5 Yield point

The exact procedure for measuring the yield point of the Raphia palm seed oil depends on the specific instrument used. However, for this research only rotational rheometer methods were employed:

Rotational Rheometer:

This is the most precise method and is often used in research settings. Here's a general outline:

1. Sample preparation: The oil sample is carefully placed within the rheometer, which typically involves a specialized container or geometry.
2. Shear rate and stress measurement: The rheometer applies a gradually increasing shear rate (force per unit area) to the oil. It then measures the resulting shear stress (deformation force) at each shear rate.
3. Data analysis: The data is plotted as a shear stress vs. shear rate curve. The yield point is not directly measured but rather calculated from the curve. Popular models like Bingham Plastic Model use the curve's intercept with the shear stress axis at zero shear rate to determine the yield point.

3.2.2.6 Cloud point

Cloud point is the temperature below which wax in liquids tends to form a cloudy appearance. It is the highest temperature at which the oil begins to solidify. To determine the cloud point of the Raphia palm seed oil (RPSO), the following steps were taken:

1. The RPSO was poured into a test tube and covered with a foil paper
2. A thermometer was inserted in it and it was placed in the freezer where at intervals of 5 minutes, it was checked to see if wax had formed
3. When it became cloudy it was taken out and the temperature was quickly read. This temperature was taken as the cloud point.

3.2.2.7 Pour Point

This is the temperature below which the liquid (RPSO) loses its flow characteristics. After the cloud point was taken, it was returned to the freezer and was checked every 5 minutes. When the test tube was tilted for 5 seconds and the oil could not flow again, the temperature was immediately read and recorded.

3.2.2.8 Acid Value Test

1. 34g of 0.05mol of potassium hydroxide (KOH) was measured in dissolved in 20ml of water.
2. The mixture was brought up to 1000 ml with ethanol to make alcoholic KOH.
3. 50ml of ethanol and 50 ml of diethyl ether was measured and mixed together. 25ml of this mixture was mixed with 2g of the RPSO.
4. After it had been mixed, 2 drops of phenolphthalein indicator were dropped in it.
5. This mixture was then titrated against the alcoholic KOH until a pink color appeared while shaking continuously.
6. This was done 3 times to get an average titre value.
7. A blank experiment was done which did not include the oil in the mixture.
8. The acid value was calculated using equation (3.5)

$$\text{Acid value} = \frac{0.1N \times Mw \times (B-V)}{Weight\ of\ oil\ used} \tag{3.5}$$

Where N is the Normality of the base, Mw is the molecular weight of KOH, B is the blank titre value and V is the average titre value.

3.2.2.9 Free Fatty Acid (%FFA) Test

The %FFA was gotten using equation (3.6)

$$\%\text{FFA} = \frac{Acid\ Value}{2} \tag{3.6}$$

3.2.2.10 Saponification Test

Saponification is the hydrolysis of esters. Oils and fats are the fatty acid esters of the trihydroxy alcohol, glycerol. The saponification value of an oil is defined as the number of milligrams of potassium hydroxide required to neutralize the fatty acids resulting from the complete hydrolysis of 1 g of the sample (Kim and Siang, 2022). The process was as follows:

1. 0.5mol of Hydrochloric acid was prepared using 50 ml of conc. Hydrochloric acid and 1000 ml of water
2. 0.5KOH was also prepared by dissolving 28.05g of KOH pellets in 1000 ml of water
3. Ethanol ether was prepared by mixing ethanol with diethyl ether.
4. 4ml of the ethanol ether was mixed with 25 ml of the KOH solution. In this mixture,1g of RPSO was mixed with it.

5. This final mixture was mixed on a magnetic heating stirrer for about 30 minutes. When it was done, it was allowed to cool, then 3 drops of phenolphthalein indicator were placed in it. The mixture turned pink in color shown in figure 3.8.
6. This was then titrated against the 0.5mol of HCl, shaking continuously, until the pink color vanished.
7. This was done 3 times to get an average titre value.
8. A blank experiment was done which did not include the oil in the mixture.
9. The saponification value was then calculated using equation (3.7)

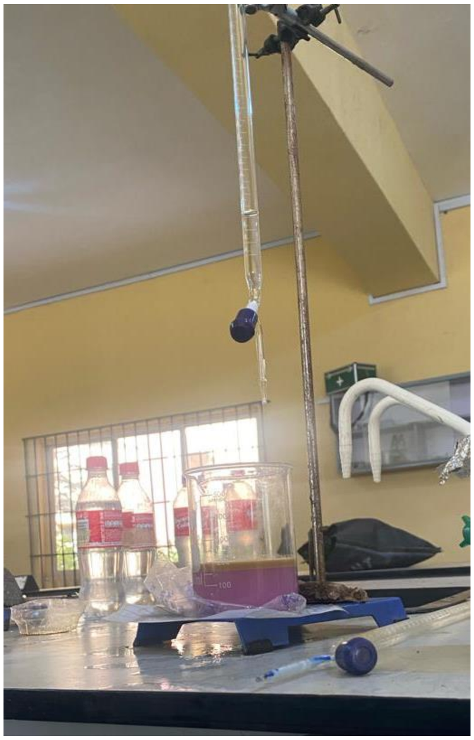


Figure 3.8: Saponification Test

$$\text{Saponification Value} = \frac{Mw \times M \times (Vb - Va)}{W} \qquad (3.7)$$

Where M is the molarity of acid, Vb is the volume of blank titre, Va is actual average titre volume and W is weight of oil used.

3.2.3 Formulation of the Raphia Palm Seed Oil Based-Mud

Drilling mud is usually a mixture of water, clay, weighing material and a few chemicals. Sometimes oil may be used instead of water, or oil added to the water to give the mud certain desirable properties. Drilling mud is used to raise the cuttings made by the bit and lift them to the surface for disposal. But one of the major functions of a drilling mud includes providing hydrostatic pressure to prevent formation fluids from entering the wellbore. To ensure that the hydrostatic pressure balances out with formation pressure and that the wellbore is stable, the mud density, along other properties, has to be taken into consideration. Higher formation pressure requires higher mud density (this can be achieved by adding more barite to the drilling mud) and vice versa. Using an incorrect drilling mud (in terms of its mud density), can result in formation damage thereby leading to a well blowout. Also, we can use other materials for preparing mud with different materials.

In this research, Raphia palm seed oil (RPSO) will be used as synthetic base oil to formulate an oil base mud which will meet the API standard requirement for oil base mud for drilling.

3.2.3.1 Procedure:

1. Weigh 245ml of RPSO into a mixer cup and mix for minutes
2. Add 6ml of primary emulsifier and 4ml of secondary emulsifier
3. Add 105ml of water and mix to homogeneity with mixing for 5 minutes
4. Add 25g of Bentonite and agitate for 10 minutes
5. Add 10g of Barite and mix for 5 minutes
6. Add 0.25g of PAC-R, mix for 5 minutes
7. Add 0.25g of CMC, mix for 5 minutes
8. Add 0.25g of NaCO_3, mix for 5 minutes
9. Add 0.25g of NaOH, mix for 5 minutes
10. Steps 1 to 9 were repeated for the other sample (diesel) as control for this research.

3.2.4 Characterization of the Raphia Palm seed oil base Mud with the Conventional Oil base mud (Diesel)

The density of the base fluids (Raphia palm seed oil and diesel) was measured using the mud balance.

a. Using the weighing balance, the various quantities of materials as shown in figure 3.9 below were measured.

Figure 3.9: Weighing balance measuring Bentonite

b. The quantities of oil were measured using measuring beakers.

c. Using the Hamilton mud mixer, the measured materials were thoroughly mixed until a homogenous mixture was obtained.

d. The mud samples (RPSO and diesel based) were aged for 24 hours.

3.2.4.1 Determination of Mud Density.

1. The aged mud samples (RPSO and diesel based) were agitated for 2 minutes using the spatula.
2. The clean, dry mud balance cup was filled to the top with the newly agitated mud.
3. The lid was placed on the cup and the balance was washed and wiped clean of overflowing mud while covering the hole in the lid.
4. The balance was placed on a knife edge and the rider moved along the arm until the cup and arm were balanced as indicated by the bubble.
5. The mud weight was read at the edge of the rider towards the mud cup as indicated by the arrow on the rider and was recorded.
6. Steps 1 to 5 were repeated for the other sample (diesel).

Figure 3.10: Mud Balance

3.2.4.2 Determination of Mud Viscosity.

7. Collect a fresh mud sample (RPSO and diesel based).
8. Hold the funnel erect with a finger over the outlet tube, and pour the mud into the funnel through the screen until the mud level reaches the bottom of the screen (The screen will filter out the larger particles that could clog the outlet tube).
9. Note when the Marsh Funnel is filled to the proper level it holds more than one quart of mud.
10. Quickly remove the finger from the outlet tube, and at the same time, begin timing the mud outflow.
11. Allow one quart (946 cc) of mud to drain from the Marsh Funnel into a graduated container.
12. Record the number of seconds it takes for the quart of mud to flow out of the funnel, and report this value as the Marsh Funnel Viscosity. Also record the temperature of the mud sample in degrees F or C.
13. Steps 7 to 10 were repeated for the other sample (diesel).

Figure 3.11: Marsh Funnel viscometer

3.2.4.3 Determination of Mud Filtration Properties.

14. The assembly is a shown in figure 3.11.
15. Each part of the cell was cleaned, dried and the rubber gaskets were checked.
16. The cell was assembled as follows: base cap, rubber gasket, screen, filter paper, rubber gasket and cell body.

Figure 3.12: API Filter Press.

17. A freshly stirred sample of mud (RPSO) was poured into the cell to within 0.5 inch (13 millimeters) to the top in order to minimize contamination of the filtrate. The top cap was checked to ensure that the rubber gasket was in place and seated all the way around and

completed the assembly. The cell assembly was placed into the frame and secured with the T-screw.

18. A clean dry graduated glass cylinder was placed under the filtrate exit tube.
19. The regulator T-screw was turned counterclockwise until the screw was in the right position and the diaphragm pressure was relieved. The safety bleeder valve on the regulator was put in the closed position.
20. The air hose was connected to the designated pressure source. The valve on the pressure source was opened to initiate pressurization into the air hose. The regulator was adjusted by turning the T-screw clockwise so that a pressure was applied to the cell in 30 seconds or less. The test period begins at the time of initial pressurization.
21. At the end of 30 minutes the volume of filtrate collected was measured. The air flow through the pressure regulator was shut off by turning the T-screw in a counterclockwise direction. The valve on the pressure source was then closed and the relief valve was carefully opened.
22. The assembly was then dismantled, and the mud was removed from the cup.
23. The filter cake was measured using a vernier caliper, and the measurements were recorded.
24. The above approach was repeated for the other sample (diesel).

3.2.4.4 Determination of Mud Hydrogen ion concentration (pH) – pH meter

25. Make sure that the meter is set to the pH Mode and adjust the temperature to 25°C.
26. Place the electrode in the sample (RPSO) to be tested.
27. Take the temperature reading of the sample.
28. The pH of the solution appears in the display.
    NOTE: Allow the display to stabilize before taking your reading!
29. Rinse the pH electrode and place it back in the storage solution.
30. The above approach was repeated for the other sample (diesel).

3.2.4.5 Determination of Mud Sand Content

31. Pour the sample (RPSO) into the sand content tube until it fills up to the mark labeled "Mud to Here."

32. Add water to the mark labeled "Water to Here." Add oil instead of water when testing oil-base mud.
33. Cover the mouth of the tube and shake vigorously.
34. Pour this mixture through the screen.
35. Add more oil, for oil-base mud to the tube, shake, and pour it through the same screen.
36. Gently wash the sand retained on the screen with a stream of oil, for oil-base mud to remove all mud and shale particles.
37. Fit the funnel upside-down over the top of the screen.
38. Turn the tip of the funnel into the mouth of the washed tube.
39. Wash the sand back into the tube with oil with oil-base mud applied to the back of the screen.
40. Allow the sand to settle in the tube and read the volume percent of sand
41. Steps 31 to 40 were repeated for the other sample (diesel).

3.2.4.6 Determination of the Toxicity Level of the Mud.

42. After the oil base mud samples (RPSO and diesel based) have been formulated, each is then tested on living organisms (plants), to see the effects on the living organisms. Base fluids; diesel and RPSO, the survival rate was measured, and the number of days of survival. The seeds exposed to RPSO survived for 6 days, while that exposed to diesel mud survived for 2 days and then withered. When the soil was checked, there was no sign of any living organisms in diesel mud sample while that of the RPSO mud, there were signs of some living organisms such as earth worms, and other little insects.

Figure 3.13: Diagram for Toxicity test

CHAPTER FOUR

4.0 RESULTS AND DISCUSSIONS

4.1 Results

This includes the values gotten from the extraction of Raphia palm seed oil (RPSO), physiochemical properties of RPSO, production and formulation of Raphia Palm Seed oil base mud, characterization of the RPSO with conventional diesel base mud, biodiesel production and characterization.

4.1.1 Results from physiochemical properties characterization

4.1.1.1 Characterization of the Raphia Palm seed oil

Using equation 3.1, % oil yield = 41.18%

Table 4.1: Raphia Palm Seed Oil (RPSO) properties

S/N	Physiochemical properties	Values
1	Density (g/cm^3)	0.834
2	Kinematic Viscosity at 40℃ (cSt)	0.773
3	Cloud Point (℃)	-10
4	Pour point ℃	-14
5	Ph	9.40
6	Acid value (mgKOH/g)	20.18
7	Free fatty acid (%)	10.09
8	Specific Gravity	0.834
9	Saponification value (mgKOH/g)	0.698

4.1.2 Characterization of the Raphia Palm seed oil base Mud with the Conventional Oil base mud (Diesel)

Table 4.2: API Standard for Oil based mud

S/N	Tests	Values
1	Density (ppg)	8.65 - 9.60
2	Specific Gravity	
3	Viscosity(secs/quartz)	52 – 56
4	Temperature ℃	27
5	Ph	9.5 – 12.5
6	Filtration Loss at 30mins (ml)	<4
7	Toxicity (days)	
8	Sand Content (%)	1 – 2

4.1.2.1 Characterization of the Raphia Palm seed oil base Mud

Table 4. 3: Raphia Palm Seed Oil (RPSO) Mud

S/N	Tests	Values
1	Density (ppg)	8.58
2	Specific Gravity	1.00
3	Viscosity(secs/quartz)	213
4	Temperature ℃	30.7
5	pH	10.01
6	Filtration Loss at 30mins (ml)	4.4
7	Toxicity (days)	6
8	Sand Content (%)	0.09

4.1.2.2 Characterization of the Diesel oil base Mud

Table 4. 4: Diesel Oil base Mud

S/N	Tests	Values
1	Density (ppg)	8.32
2	Specific Gravity	0.98
3	Viscosity(secs/quartz)	315
4	Temperature ℃	28.1
5	pH	11.45
6	Filtration Loss at 30mins (ml)	9
7	Toxicity (days)	2
8	Sand Content (%)	0.2

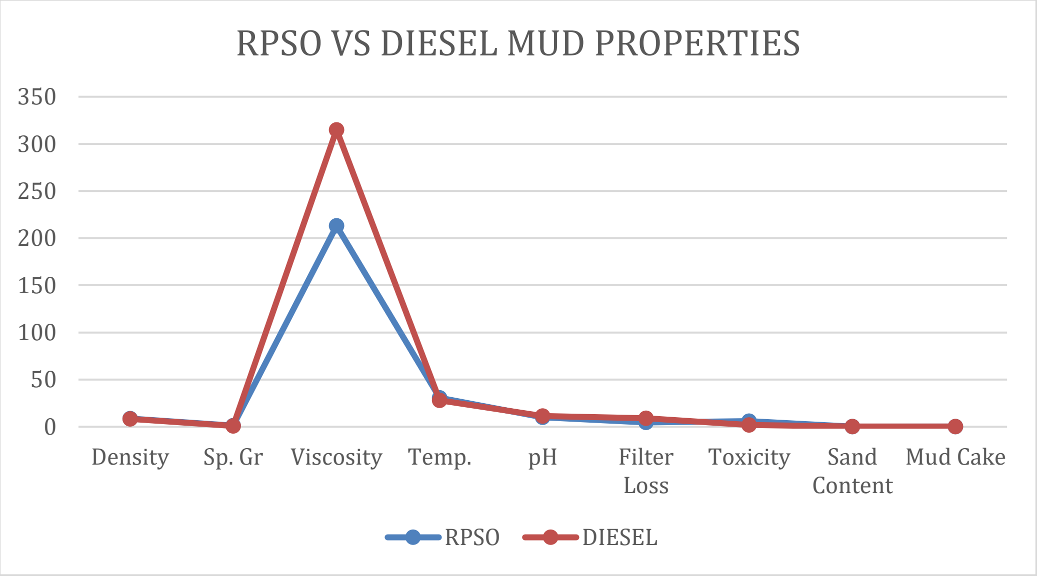


Figure 4.1: Plot of the Mud properties of RPSO vs Diesel

4.1.3 FAME Production and Physiochemical characterization

4.2 Discussion

4.2.1 Determination of Mud Density

Tests were conducted using Raphia Palm Seed Oil (RPSO) and diesel as base oils for drilling mud. The RPSO mud exhibited a higher density (8.58 ppg) compared to diesel mud (8.32 ppg). Since the recommended mud density by the API standard is 8.65 ppg, RPSO emerges as a more suitable base oil due to its closer density match.

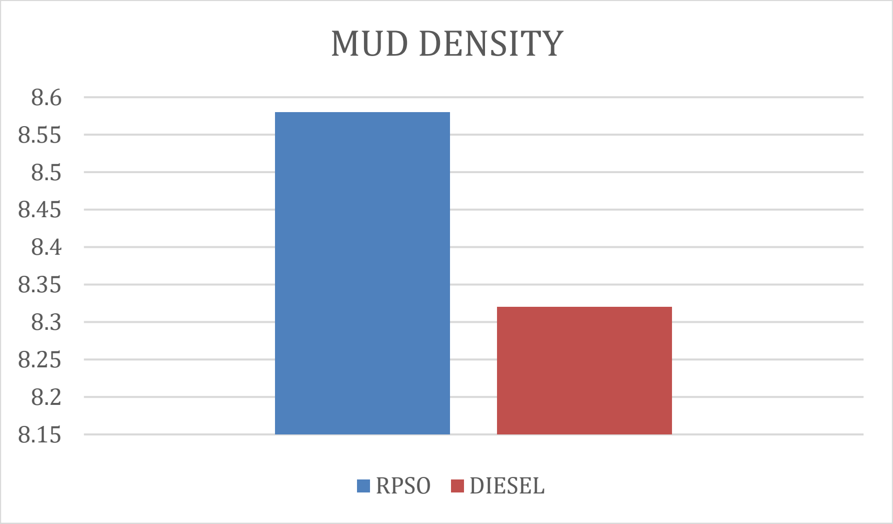


Figure 4.2: Comparison between the Mud Densities of RPSO and Diesel

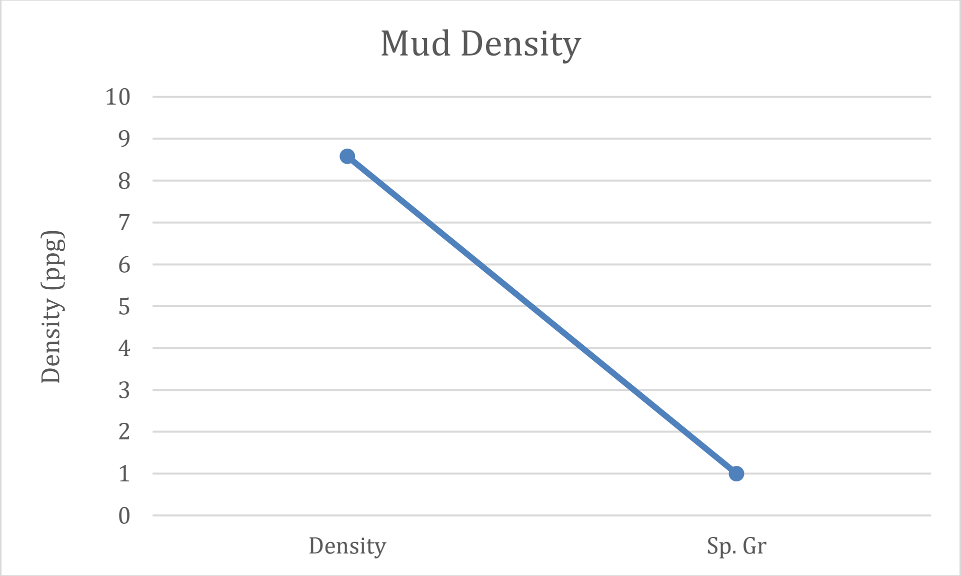


Figure 4.3: Plot of the Mud Density of RPSO vs Diesel

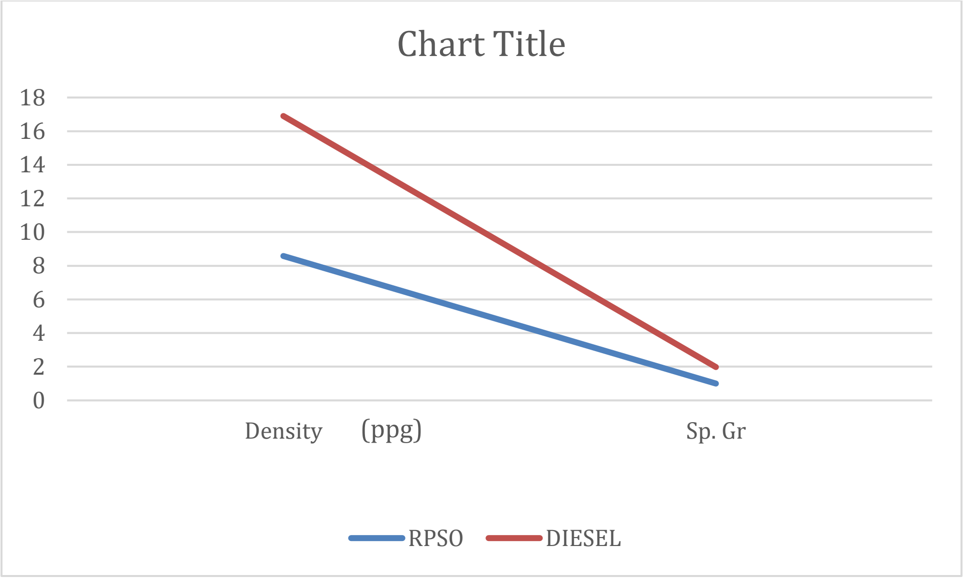


Figure 4.4: Comparison between the Mud Density and Specific Gravity of RPSO and Diesel

4.2.2 Determination of Mud Viscosity

Using the marsh funnel Viscometer, the viscosities of both oil-based drilling mud (RPSO and Diesel) was obtained as 213 and 315 in secs/quartz and a at temperature of 30.7 and 28.1 in ℃ respectively. It can be deduced that the RPSO drilling mud is less viscous than the Diesel mud and both samples are similar to the Bingham plastic model. This goes to prove that the muds have similar rheological behavior. A Bingham plastic fluid will not flow until the shear stress τ exceeds a certain minimum value (τy) known as the yield point (Bourgoyne et al 1991). After the yield has been exceeded, the changes in shear stress are proportional to changes in shear rate and the constant of proportionality is known as the plastic viscosity (μp). For reduced friction during drilling, Raphia Palm OBM gives the best results. This means Diesel OBM offers the greatest resistance to fluid flow. Raphia Palm OBM poses a better prospect in the sense that its lower viscosity will mean less resistance to fluid flow. This will in turn lead to reduced wear in the drill string.

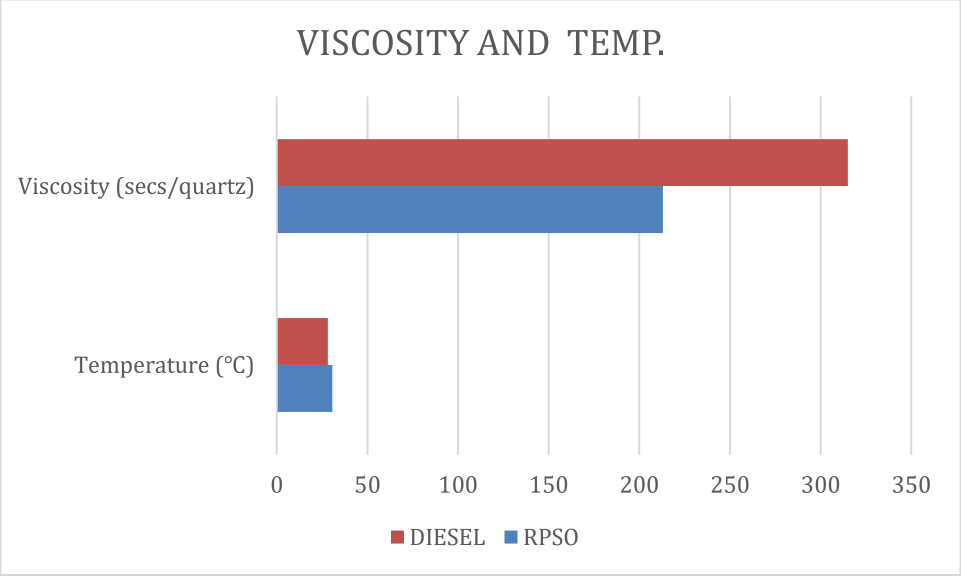


Figure 4.5: Comparison between Viscosity and Temperature of RPSO and Diesel

4.2.3 Determination of Mud Filtration Properties

From the results of the filtration test we can infer that Diesel OBM had the highest rate of filtration and spurt loss. Comparing this to a drilling scenario, this means that the mud cake from Diesel OBM is the most porous and the thickest. From these inferences, we can see that RPSO OBM is better in filtration properties than Diesel OBM as inferred from thickness and filtration volumes. Problems caused as a result of excessive thickness include:

1. Tight spots in the hole that cause excessive drag.
2. Increased surges and swabbing due to reduced annular clearance.
3. Differential sticking of the drill-string due to increased contact area and rapid development of sticking forces caused by higher filtration rate.
4. Primary cementing difficulties due to inadequate displacement of filter cake.
5. Increased difficulty in running casing.

The problems as a result of excessive filtration volumes include:

1. Formation damage due to filtrate and solids invasion and damaged zone too deep to be remedied by perforation or acidization. Damage may be precipitation of insoluble compounds, changes in wettability, and changes in relative permeability to oil or gas, formation plugging with fines or solids, and swelling of clays.

2. Invalid formation-fluid sampling test. Formation-fluid flow tests may give results for the filtrate rather than for the reservoir fluids.
3. Formation-evaluation difficulties caused by excessive filtrate invasion, poor transmission of electrical properties through thick cakes, and potential mechanical problems running and retrieving logging tools.
4. Erroneous properties measured by logging tools (measuring filtrate altered properties rather than reservoir fluid properties).
5. Oil and gas zones may be overlooked because the filtrate is flushing hydrocarbons away from the wellbore, making detection more difficult.

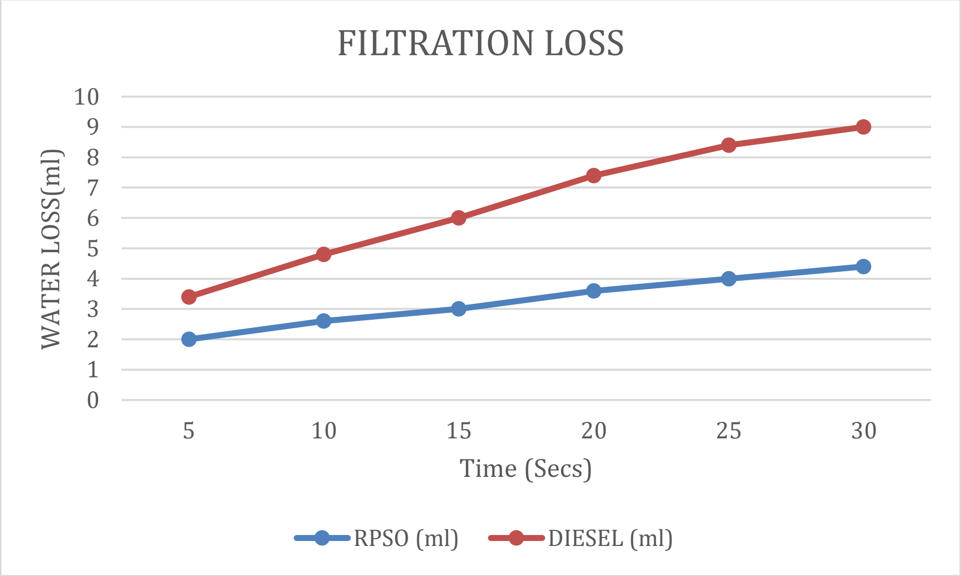


Figure 4.6: Comparison between the Mud Filtration Loss of RPSO and Diesel

<u>Mud Cake:</u>

Table 4.5: Mud Cake values of RPSO and Diesel after Filtration Loss

S/N		RPSO	DIESEL
1	MUD CAKE (%)	0.15	0.2

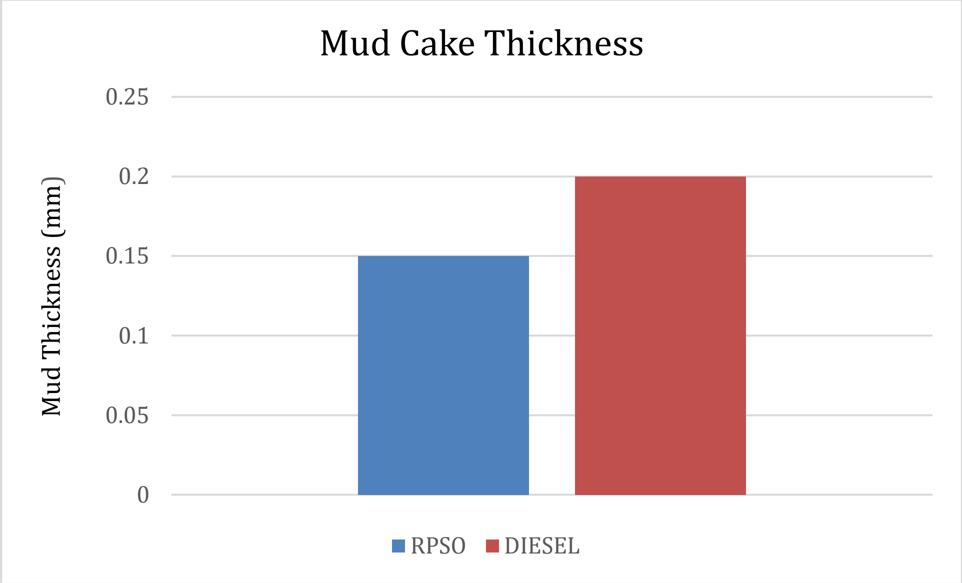


Figure 4.7: Comparison between the Mud Cake Thickness of RPSO and Diesel

4.2.4 Determination of Hydrogen ion concentration (Mud pH)

Drilling muds are always treated to be alkaline (i.e., a pH greater than 7). The pH will affect viscosity, bentonite is least affected if the pH is in the range of 7 to 9.5. Above this, the viscosity will increase and may give viscosities that are out of proportion for good drilling properties. For minimizing shale problems, a pH of 9.5 to 12.5 appears to give the best hole stability and control over mud properties. A high pH appears to cause shale problems. The corrosion of metal is increased if it comes into contact with an acidic fluid. From this point of view, the higher pH would be desirable to protect pipe and casing (Baker Hughes, 1995). The pH values of the samples meet a few of the requirements stated. Both RPSO and Diesel OBM shows better result since their pH values falls within this range (10.01 and 11.45) respectively.

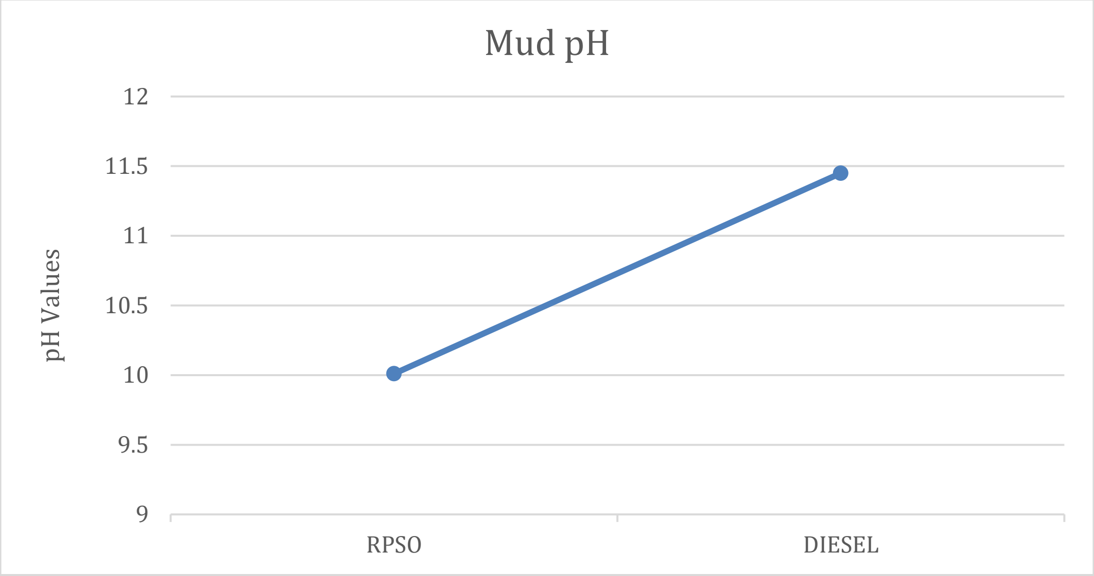


Figure 4.8: Comparison between the Mud Hydrogen ion concentration of RPSO and Diesel

4.2.5 Determination of the Toxicity Level of the Mud.

From the results of the toxicity test, it can be concluded that Raphia Palm oil-based mud has less harmful effect on plant growth compared to diesel oil-based mud. This shows that RPSO mud sample is environmentally safer for both plants and micro animals than diesel mud sample. Biodegradation and bioaccumulation however depend on the chemistry of the molecular character of the base fluids used. In general, green material i.e., plant materials containing oxygen within their structure degrade easier.

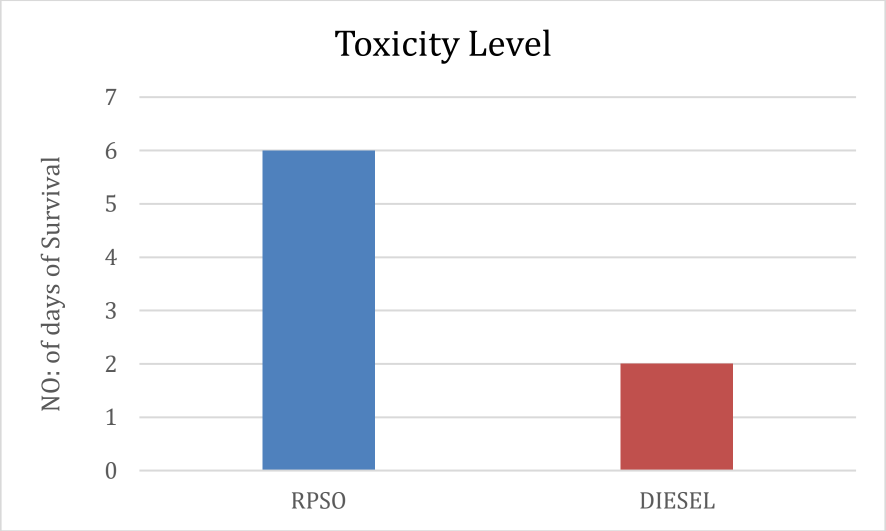


Figure 4.9: Comparison between the Mud Toxicity Level of RPSO and Diesel

4.2.6 Determination of Mud Sand Content

Regular determination of the sand content of drilling mud is necessary because these particles can be highly abrasive, and can cause excessive wear of pump parts, drill bits, and pipe connections, excessive sand may also result in the deposition of a thick filter cake on the walls of the hole, or it may settle in the hole around the tools when circulation is temporarily halted, interfering with the operation of drilling tools of settling casing. The sand content test for set is used in the test for sand content determination using Baroid sand content set. Excessive sand may also result in the deposition of a thick filter cake on the borehole wall, or it may settle in the hole around the tools when circulation is stopped. From the test carried out, Raphia Palm Seed OBM has the most favorable sand content value (0.09%) for drilling operation.

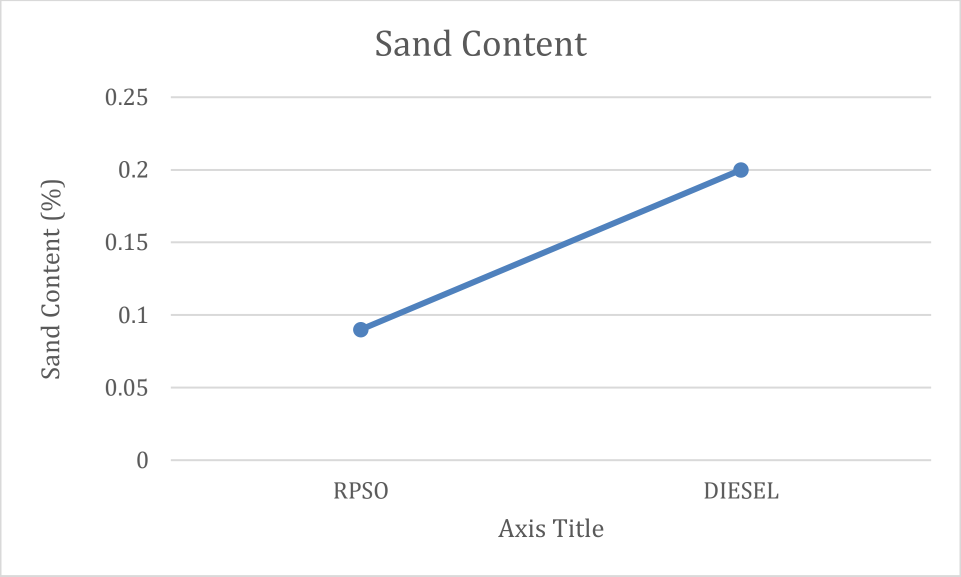


Figure 4.10: Comparison between the Mud Sand Content of RPSO and Diesel

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

From the results obtained, the similarity in the characteristics of the Raphia palm seed oil (RPSO) and the conventional diesel oil has proved that RPSO can be used as a viable substitute for the conventional diesel oil in the formulation of oil-based drilling mud. Therefore, attention has to be focused on the use of oil from non-edible plant seeds for drilling mud production instead of edible oils. The results of the tests carried out indicate that Raphia palm seed OBM has a great chance of being among the technically viable replacement of diesel OBM. The results also show that additive chemistry must be employed in the mud formulation, to make them more technically feasible.

The tests of temperature effects on density: The densities increased and became constant at some point, and began increasing again (these temperature points of constant density varied for the different samples). The diesel OBM showed the highest variation range, while the Raphia palm seed OBM showed the lowest.

5.2 Recommendations

This work should further be tested and investigated for the effect of temperature on other properties of the formulated drilling fluids.

The temperature-density tests should also be carried out at varying pressures, to simulate downhole conditions.

5.3 Contribution to knowledge

This study has proven to have the potential to introduce innovative solutions to the oil and gas industry for the purpose of drilling by demonstrating the feasibility and effectiveness of Raphia palm seed-based oil muds. Such innovations are crucial for fostering a shift towards sustainable practices and garnering industry-wide adoption. The research aligns with broader global sustainability goals, such as those outlined in the United Nations' Sustainable Development Goals (SDGs). Addressing environmental concerns in the oil and gas industry through sustainable drilling practices supports these international initiatives.

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APPENDIX

FOR RAPHIA PALM SEED OIL:

1. **Oil Yield**

Using equation 3.1 to calculate % oil yield,

$$\% \text{ oil yield} = \frac{Weight\ of sample\ before\ extraction - Weight\ of\ sample\ after\ extraction}{weight\ of\ sample\ before\ extraction} \times 100\% \qquad (A.1)$$

$$\% \text{ Oil yield} = \frac{5703.63 - 3354.88 (in\ grams)}{5703.63\ (in\ grams)} \times 100\% \quad = 41.18\%$$

2. **Density**

Weight of empty pycnometer = 23.03g

Weight of pycnometer filled with distilled water = 50.47g

Weight of pycnometer with oil = 45.92g

Weight of oil = 200.96g

a = 50.47-23.03 = 27.44g

b = density of water in g/cm^3 = $1g/cm^3$

$$\text{Volume of water} = \frac{27.44g}{1g/cm^3} = 27.44cm^3 \qquad (A.2)$$

$$\text{Density of oil } (\rho) = \frac{mass\ of\ oil}{volume\ of\ reference\ fluid (water)} \qquad (A.3)$$

$$(\rho) = \frac{45.92 - 23.03}{27.44} = \frac{22.89}{27.44} = 0.834\ g/cm^3$$

3. **Specific Gravity**

$$\text{Specific gravity of oil } (\gamma) = \frac{density\ of\ oil\ extracted}{density\ of\ water} \qquad (A.4)$$

$$(\gamma) = \frac{0.834 g/cm^3}{1g/cm^3} = 0.834$$

4. **Viscosity**

Dynamic Viscosity (DV) = 6.45cp

Kinematic Viscosity = ((DV) × spindle factor)/density (A.5)

= (6.45 × 0.1)/0.834 = 0.773cSt

5. **Acid Value**

$$\text{Acid value} = \frac{0.1N \times Mw \times (B-V)}{Weight\ of\ oil\ used} \qquad (A.6)$$

Molecular weight of KOH (Mw) = 56.1

Blank titre (B) = 147ml

Average titre (V) = 144ml

= 0.1×56.1× (147 - 144)/200.96

Acid value = 20.180mgKOH/g

6. **Free Fatty Acid (%FFA) Test**

$$\%FFA = \frac{Acid\ Value}{2} \qquad (A.7)$$

$$\%FFA = \frac{20.18}{2} = 10.09\%$$

7. **Saponification Value**

$$\text{Saponification Value} = \frac{Mw \times M \times (Vb-Va)}{W} \qquad (A.8)$$

Average titre = 144ml Blank titre = 149ml

56.1×0.5 × (149 - 144)/200.96 = 0.698mgKOH/g